



(12) **United States Patent**
Bertagnolli et al.

(10) **Patent No.:** **US 8,616,306 B2**
(45) **Date of Patent:** ***Dec. 31, 2013**

(54) **POLYCRYSTALLINE DIAMOND COMPACTS, METHOD OF FABRICATING SAME, AND VARIOUS APPLICATIONS**

(58) **Field of Classification Search**
USPC 175/420.2, 328, 428, 433, 434; 51/293
See application file for complete search history.

(71) Applicant: **US Synthetic Corporation**, Orem, UT (US)

(56) **References Cited**

(72) Inventors: **Kenneth E. Bertagnolli**, Riverton, UT (US); **Michael A. Vail**, Glenola, UT (US); **Jason K. Wiggins**, Draper, UT (US); **Jiang Qian**, Cedar Hills, UT (US); **David P. Miess**, Highland, UT (US); **Debkumar Mukhopadhyay**, Sandy, UT (US)

U.S. PATENT DOCUMENTS

3,101,266 A 8 1963 Cheney
3,141,746 A 7 1964 De Lai

(Continued)

FOREIGN PATENT DOCUMENTS

EP 0462955 12 1991
JP 121251 6 1985

(Continued)

OTHER PUBLICATIONS

U.S. Appl. No. 12/690,998, Oct. 10, 2012, Issue Notification.

(Continued)

Primary Examiner Cathleen Hutchins

(74) *Attorney, Agent, or Firm* Dorsey & Whitney LLP

(*) **Notice:** Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.
This patent is subject to a terminal disclaimer.

(21) Appl. No.: **13/623,764**

(22) Filed: **Sep. 20, 2012**

(65) **Prior Publication Data**

US 2013/0015001 A1 Jan. 17, 2013

Related U.S. Application Data

(63) Continuation of application No. 12/690,998, filed on Jan. 21, 2010, now Pat. No. 8,297,382, which is a continuation-in-part of application No. 12/244,960, filed on Oct. 3, 2008, now Pat. No. 7,866,418.

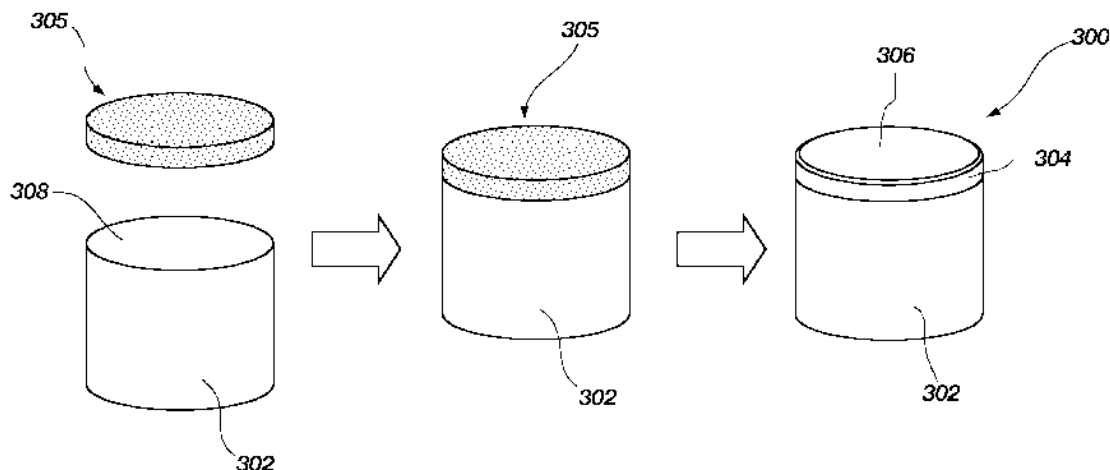
(51) **Int. Cl.**
E21B 10/36 (2006.01)
E21B 10/46 (2006.01)

(52) **U.S. Cl.**
CPC **E21B 10/36** (2013.01)
USPC **175/420.2; 175/328; 175/428; 175/433; 175/434**

(57) **ABSTRACT**

Embodiments of the invention relate to a polycrystalline diamond compact. In an embodiment, the polycrystalline diamond compact includes a substrate and a polycrystalline diamond table including a first polycrystalline diamond layer bonded to the substrate and at least a second polycrystalline diamond layer. At least an un-leached portion of the polycrystalline diamond table includes a plurality of diamond grains defining a plurality of interstitial regions and a metal-solvent catalyst occupying at least a portion of the plurality of interstitial regions. The plurality of diamond grains and the metal-solvent catalyst collectively exhibit a coercivity of about 115 Oe or more and a specific magnetic saturation of about 15 G·cm³/g or less. The second polycrystalline diamond layer exhibits a second average diamond grain size that is less than a first average diamond grain size of the first polycrystalline diamond layer and/or the first polycrystalline diamond layer includes a tungsten-containing material therein.

31 Claims, 12 Drawing Sheets



US 8,616,306 B2

Page 2

(56)

References Cited

U.S. PATENT DOCUMENTS

3,574,580 A 4 1971 Stromberg et al.
 4,425,315 A 1 1984 Isuji et al.
 4,604,106 A 8 1986 Hall et al.
 4,610,600 A 9 1986 Bleier
 4,610,699 A 9 1986 Yazu et al.
 4,636,253 A 1 1987 Nakai et al.
 4,643,741 A 2 1987 Yu et al.
 4,694,918 A 9 1987 Hall
 4,729,440 A 3 1988 Hall
 4,811,801 A 3 1989 Salesky et al.
 5,355,969 A 10 1994 Hardy et al.
 5,370,195 A 12 1994 Keshavan et al.
 5,469,927 A 11 1995 Griffin
 5,605,199 A 2 1997 Newton
 5,769,176 A 6 1998 Sumiya et al.
 5,875,862 A 3 1999 Jurewicz et al.
 5,889,219 A 3 1999 Moriguchi et al.
 6,090,343 A 7 2000 Kear et al.
 6,132,675 A 10 2000 Corrigan et al.
 6,227,318 B1 5 2001 Siracki
 6,241,035 B1 6 2001 Portwood
 6,290,008 B1 9 2001 Portwood et al.
 6,338,754 B1 1 2002 Cannon et al.
 6,342,301 B1 1 2002 Yoshida et al.
 6,443,248 B2 9 2002 Yong et al.
 6,460,637 B1 10 2002 Siracki et al.
 6,655,234 B2 12 2003 Scott
 6,749,033 B2 6 2004 Griffin et al.
 6,913,633 B2 7 2005 Fries et al.
 6,915,866 B2 7 2005 Middlemiss
 6,987,318 B2 1 2006 Sung
 7,108,598 B1 9 2006 Galloway
 7,216,661 B2 5 2007 Welty et al.
 7,350,601 B2 4 2008 Belnap et al.
 7,435,478 B2 10 2008 Keshavan
 7,462,003 B2 12 2008 Middlemiss
 7,493,972 B1 2 2009 Schmidt et al.
 7,516,804 B2 4 2009 Vail
 7,517,589 B2 4 2009 Eyre
 7,543,662 B2 6 2009 Belnap et al.
 7,575,805 B2 8 2009 Achilles et al.
 7,628,234 B2 12 2009 Middlemiss
 7,740,673 B2 6 2010 Eyre
 7,757,791 B2 7 2010 Belnap et al.
 7,866,418 B2 1 2011 Bertagnolli et al.
 8,020,645 B2 9 2011 Bertagnolli et al.
 8,158,258 B2 4 2012 Bertagnolli et al.
 8,197,936 B2 6 2012 Keshavan
 2004 0062928 A1 4 2004 Raghavan et al.
 2004 0140132 A1 7 2004 Middlemiss
 2005 0050801 A1 3 2005 Cho et al.
 2005 0139397 A1 6 2005 Achilles et al.
 2005 0210755 A1 9 2005 Cho et al.
 2005 0262774 A1 12 2005 Eyre et al.
 2006 0038156 A1 2 2006 Welty et al.
 2006 0162969 A1 7 2006 Belnap et al.
 2006 0165993 A1 7 2006 Keshavan
 2006 0180354 A1 8 2006 Belnap et al.
 2007 0014965 A1 1 2007 Chodolka et al.
 2008 0022806 A1 1 2008 Sumiya
 2008 0023231 A1 1 2008 Vail
 2008 0115424 A1 5 2008 Can et al.
 2008 0142276 A1 6 2008 Griffio et al.
 2008 0178535 A1 7 2008 Wan
 2008 0185189 A1 8 2008 Griffio et al.
 2008 0206576 A1 8 2008 Qian et al.
 2008 0302579 A1 12 2008 Keshavan et al.
 2009 0152018 A1 6 2009 Sani
 2009 0208301 A1 8 2009 Kuroda et al.
 2010 0112332 A1 5 2010 Kuroda et al.
 2010 0186304 A1 7 2010 Burgess et al.
 2010 0225311 A1 9 2010 Bertagnolli et al.
 2010 0242375 A1 9 2010 Hall et al.
 2010 0307069 A1 12 2010 Bertagnolli et al.
 2010 0307070 A1 12 2010 Bertagnolli et al.

2010 0310855 A1 12 2010 Bertagnolli et al.
 2011 0017517 A1 1 2011 Scott et al.
 2011 0017519 A1 1 2011 Bertagnolli et al.
 2011 0031032 A1 2 2011 Mourik et al.
 2011 0031033 A1 2 2011 Mourik et al.
 2011 0031037 A1 2 2011 Bellin et al.
 2011 0042147 A1 2 2011 Fang et al.
 2011 0042149 A1 2 2011 Scott et al.
 2011 0083908 A1 4 2011 Shen et al.
 2011 0189468 A1 8 2011 Bertagnolli et al.
 2012 0261197 A1 10 2012 Micss et al.

FOREIGN PATENT DOCUMENTS

WO WO 2004 106004 12 2004
 WO WO 2006 099194 9 2006
 WO WO 2007 020518 2 2007
 WO WO 2010 039346 4 2010
 WO WO 2011 011290 1 2011
 WO WO 2011 017592 2 2011

OTHER PUBLICATIONS

U.S. Appl. No. 12 858,906, Oct. 5, 2012, Office Action.
 U.S. Appl. No. 11 545,929, filed Oct. 10, 2006, Bertagnolli, et al.
 U.S. Appl. No. 12 830,878, filed Jul. 6, 2010, Wiggins et al.
 U.S. Appl. No. 13 486,578, filed Jun. 1, 2012, Bertagnolli, et al.
 D.L. Decker, W.A. Basset, L. Merrill, H.T. Hall, and J.D. Barnett: "High-Pressure Calibration A Critical Review", J. Phys. Chem. Ref. Data, vol. 1, No. 3 (1972).
 ASTM B887-03 (2008) "Standard Test Method for Determination of Coercivity (H_{cs}) of Cemented Carbides".
 ASTM B886-03 (2008), "Standard Test Method for Determination of Magnetic Saturation (M_s) of Cemented Carbides".
 W. Utsumi, N. Toyama, S. Endo, and F.E. Fujita, "X-ray diffraction under ultrahigh pressure generated with sintered diamond anvils", J. Appl. Phys., 60, 2201 (1986).
 G. Rousse, S. Klotz, A.M. Saitta, J. Rodriguez-Carvajal, M.J. McMahon, B. Couzinet, and M. Mezouar, "Structure of the Intermediate Phase of PbTe at High Pressure", Physical Review B: Condensed Matter and Materials Physics, 71, 224116 (2005).
 Tze-Pin Lin, Michael Hood, George A. Cooper, and Redd H. Smith, Residual Stresses in Polycrystalline Diamond Compacts, J. Am. Ceram. Soc. 77[6] pp. 1562-1568 (1994).
 IDR Moyle, ER Kimmel "Utilization of magnetic saturation measurements for carbon control in cemented carbides" Dec. 1984, American Society of Metals Metals Materials Technology series 1984 ASM SC11: conference on technology advancements in cemented carbide production 8415-009.
 Akashi et al.; "Synthesis of Sintered Diamond with High Electrical Resistivity and Hardness"; J. Am. Ceram. Soc.; vol. 70, No. 10; 1987; pp. C-237-X-239.
 Bochechka et al.; "The Study of HIP-HT Interaction between Co-Base Melts and Diamond Powders"; High Pressure Chemical Engineering: 1996; p. 457.
 Ekinov et al.; "Sintering of a Nanodiamond in the Presence of Cobalt"; Inorganic Materials; vol. 45, No. 5; 2009; pp. 491-494.
 Godiek; "Message from Neil B. Godiek"; PBI, Inc. Technologies, Inc.; Oct. 2008.
 Osipov et al.; "A contribution to the study of the diamond solid state sintering"; Ceramics; vol. 49; 2003; pp. 151-157.
 Shige et al.; "Sintering of Diamond Powder Electroless-Plated with Co Metal", Science and Technology of New Diamond, pp. 251-255 (1990).
 Jardim; "A Novel Method for Obtaining Polycrystalline Diamond Cutters"; Materials Science Forum; vols. 660-661; 2010; pp. 477-482.
 German; "Particle Packing Characteristics", Metal Powder Industries Federation; pp. 101-103; 1989 (6 pages).
 International Search Report and Written Opinion from International Application No. PCT/US2009/054398 Mailed Feb. 2, 2010.
 International Search Report and Written Opinion from International Application PCT/US2010/059619 mailed Mar. 4, 2011.
 U.S. Appl. No. 12 244,960, Apr. 27, 2010, Office Action.

US 8,616,306 B2

Page 3

(56)

References Cited**OTHER PUBLICATIONS**

U.S. Appl. No. 12 244,960, Jun. 16, 2010, Office Action.
 U.S. Appl. No. 12 244,960, Sep. 27, 2010, Notice of Allowance.
 U.S. Appl. No. 12 244,960, Nov. 29, 2010, Notice of Allowance.
 U.S. Appl. No. 12 244,960, Dec. 22, 2010, Issue Notification.
 U.S. Appl. No. 12 690,998, Feb. 27, 2012, Office Action.
 U.S. Appl. No. 12 690,998, Jul. 17, 2012, Notice of Allowance.
 U.S. Appl. No. 12 785,014, Sep. 10, 2012, Office Action.
 U.S. Appl. No. 12 858,949, Dec. 23, 2010, Office Action.
 U.S. Appl. No. 12 858,949, Jun. 8, 2011, Office Action.
 U.S. Appl. No. 12 858,949, Jul. 28, 2011, Notice of Allowance.
 U.S. Appl. No. 12 858,949, Aug. 31, 2011, Issue Notification.
 U.S. Appl. No. 12 846,604, Aug. 8, 2011, Office Action.

U.S. Appl. No. 12 846,604, Feb. 27, 2012, Notice of Allowance.
 U.S. Appl. No. 12 846,604, Mar. 28, 2012, Issue Notification.
 U.S. Appl. No. 13 789,099, filed Mar. 7, 2013, Bertagnolli et al.
 U.S. Appl. No. 13 790,172, filed Mar. 8, 2013, Bertagnolli et al.
 U.S. Appl. No. 12 785,014, Feb. 5, 2013, Notice of Allowance.
 U.S. Appl. No. 13 085,689, Mar. 15, 2013, Office Action.
 U.S. Appl. No. 12 608,155, filed Oct. 29, 2009, Sani.
 U.S. Appl. No. 13 275,372, filed Oct. 18, 2011, Mukhopadhyay.
 U.S. Appl. No. 13 648,913, filed Oct. 10, 2012, Mukhopadhyay.
 U.S. Appl. No. 13 909,193, filed Jun. 4, 2013, Miess, et al.
 U.S. Appl. No. 12 785,014, May 22, 2013, Issue Notification.
 U.S. Appl. No. 12 858,906, Apr. 10, 2013, Office Action.
 U.S. Appl. No. 13 790,172, May 15, 2013, Office Action.
 U.S. Appl. No. 13 486,578, Sep. 20, 2013, Office Action.
 U.S. Appl. No. 13 790,172, Oct. 21, 2013, Office Action.
 U.S. Appl. No. 13 087,775, Sep. 20, 2013, Office Action.

U.S. Patent

Dec. 31, 2013

Sheet 1 of 12

US 8,616,306 B2

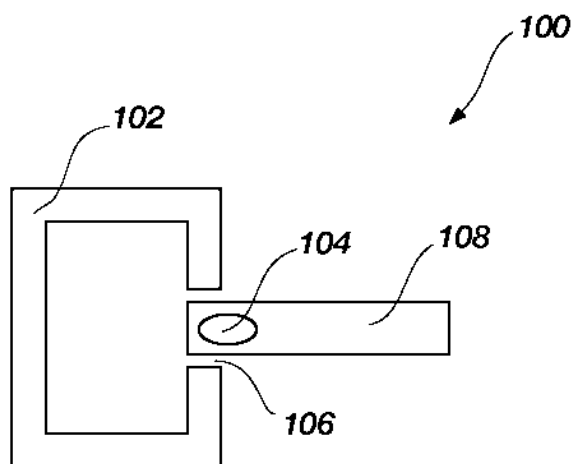


FIG. 1A

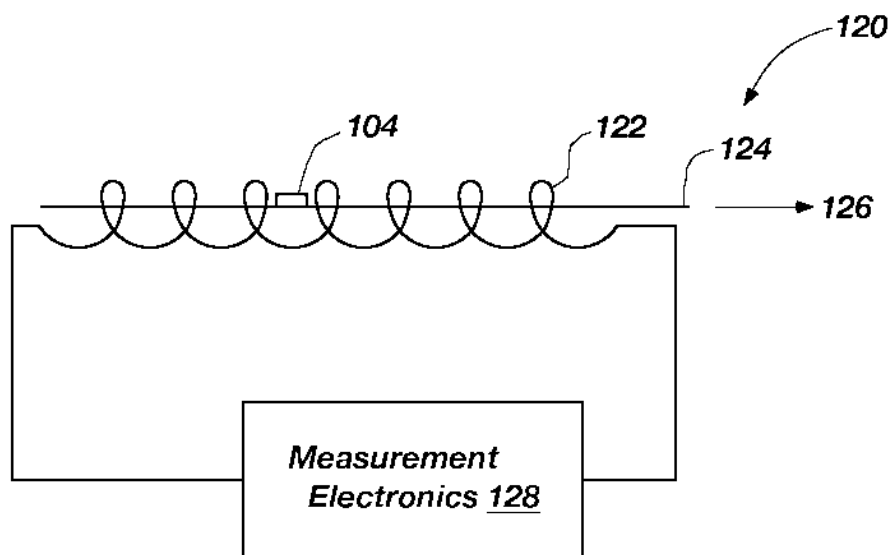


FIG. 1B

U.S. Patent

Dec. 31, 2013

Sheet 2 of 12

US 8,616,306 B2

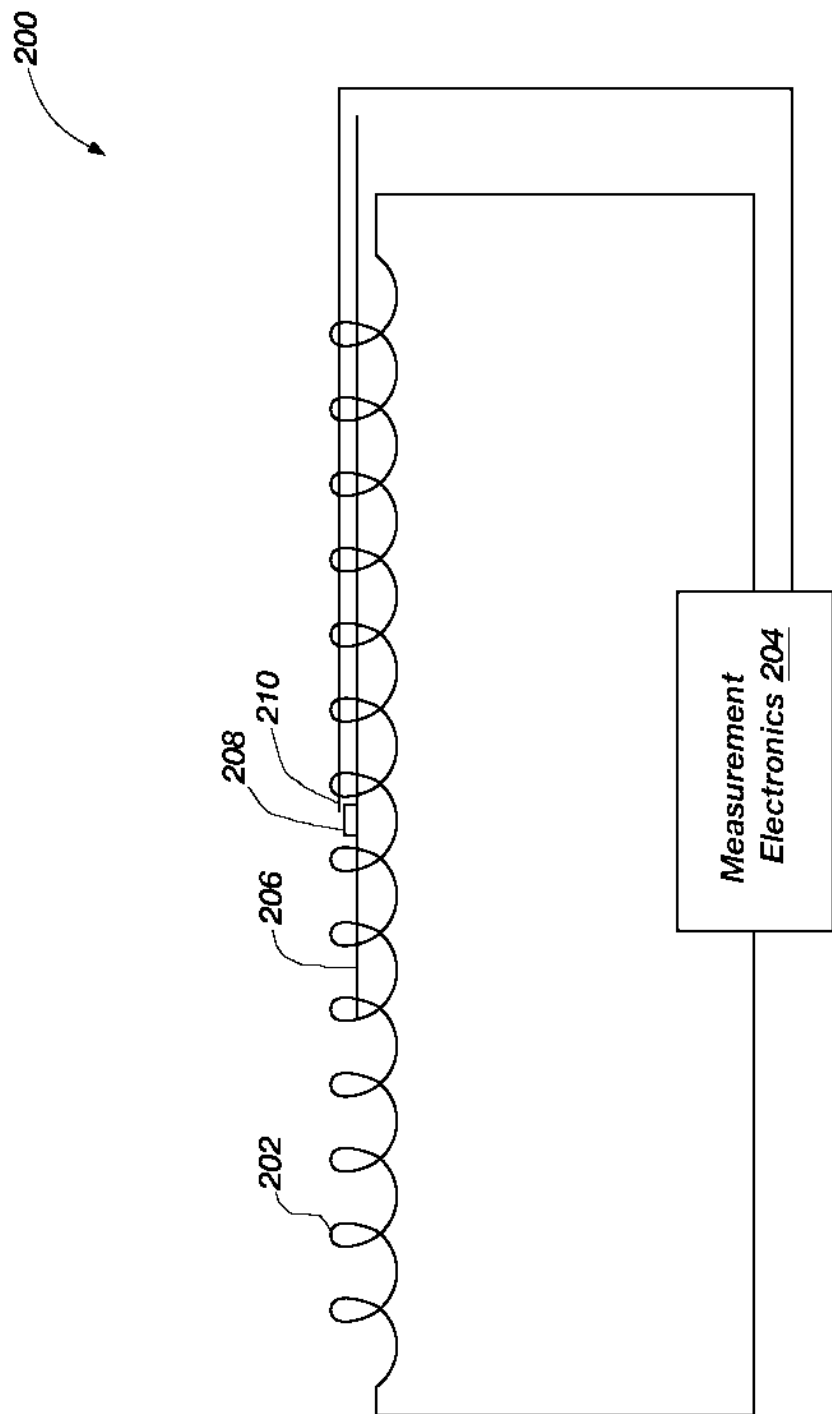


FIG. 2

U.S. Patent

Dec. 31, 2013

Sheet 3 of 12

US 8,616,306 B2

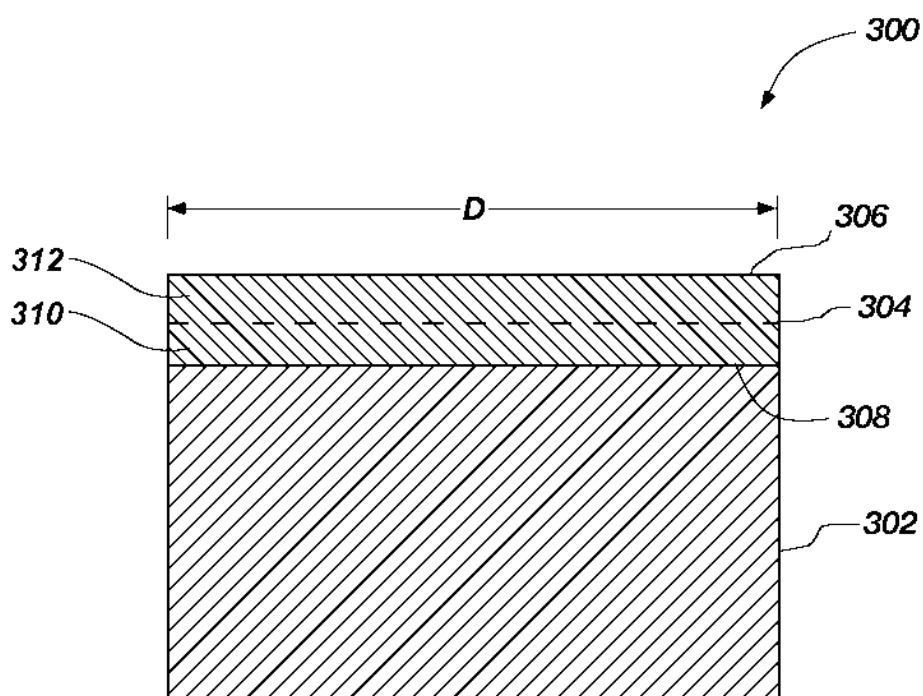
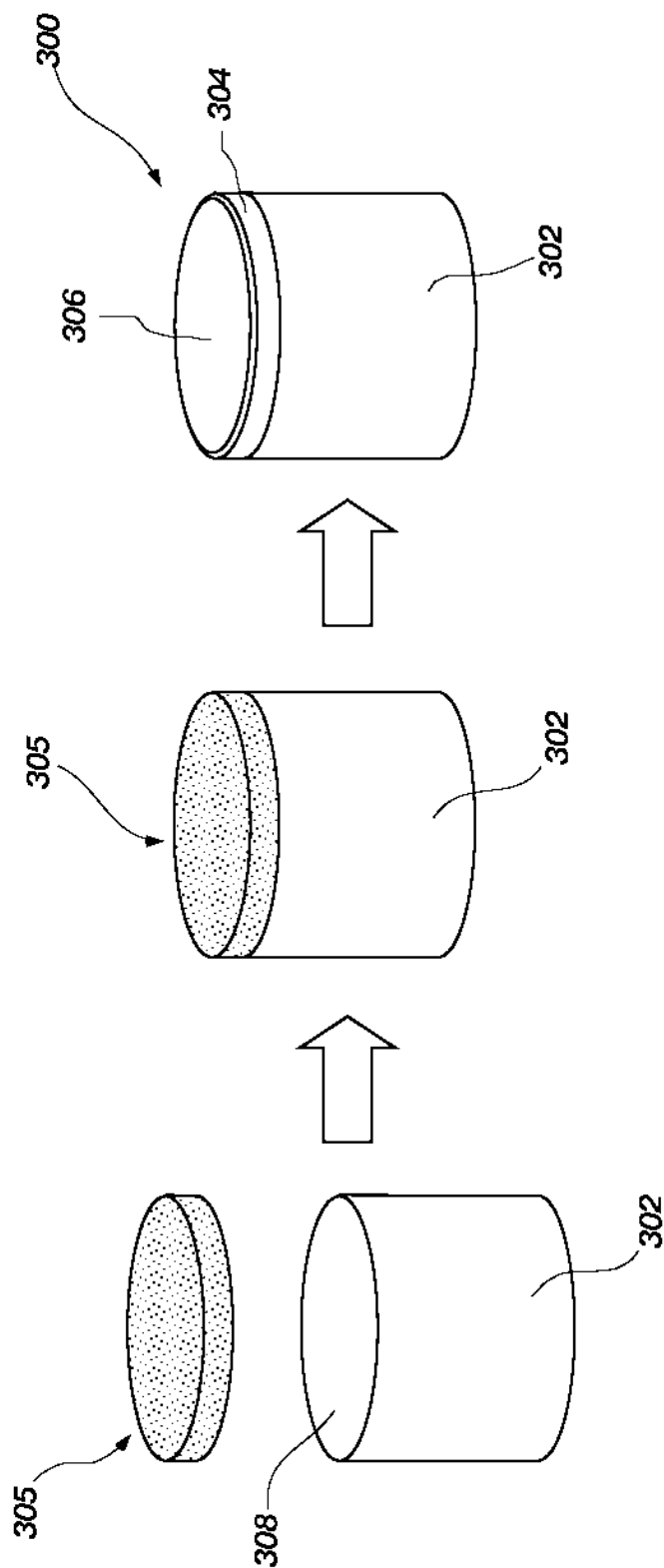


FIG. 3A



U.S. Patent

Dec. 31, 2013

Sheet 5 of 12

US 8,616,306 B2

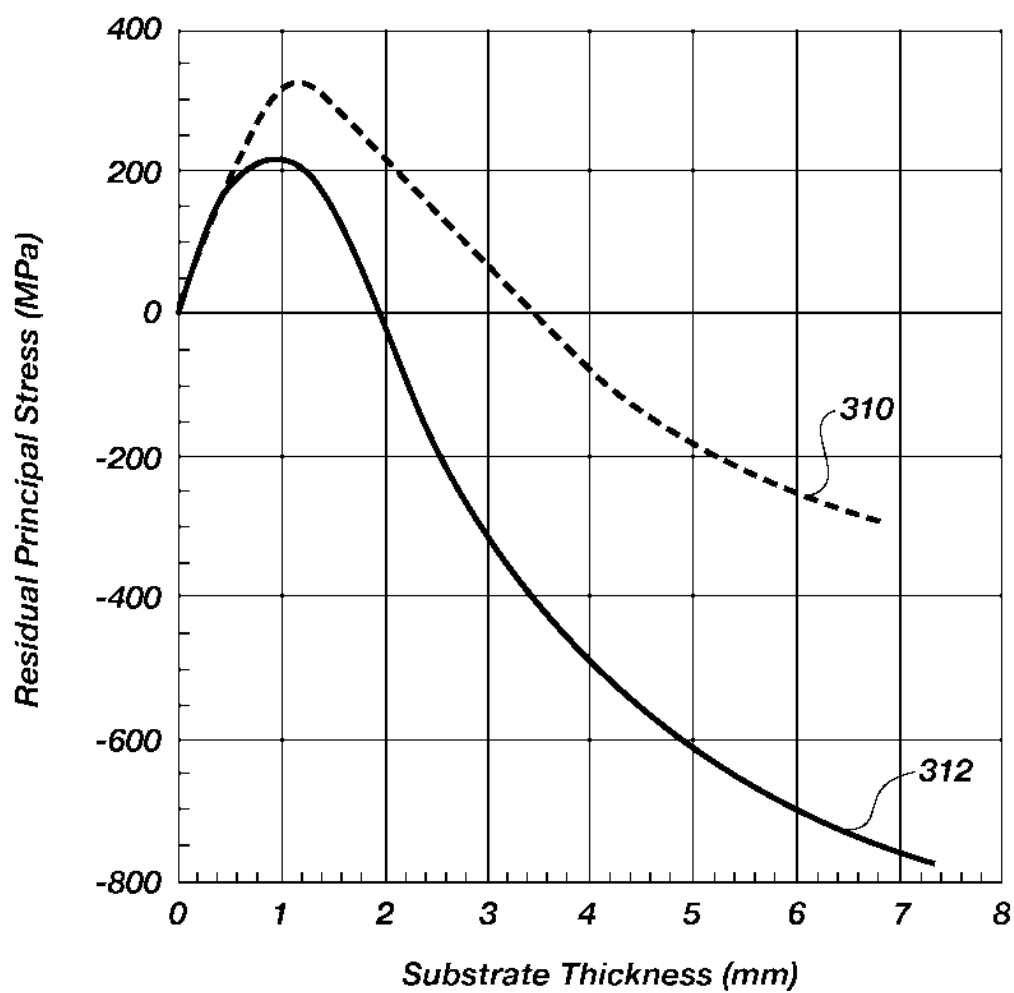


FIG. 3C

U.S. Patent

Dec. 31, 2013

Sheet 6 of 12

US 8,616,306 B2

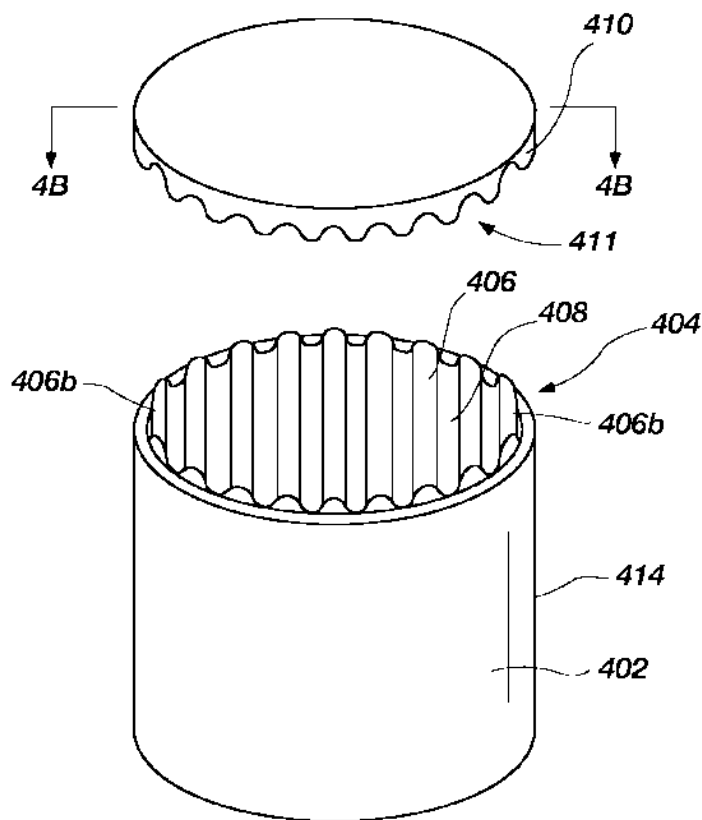


FIG. 4A

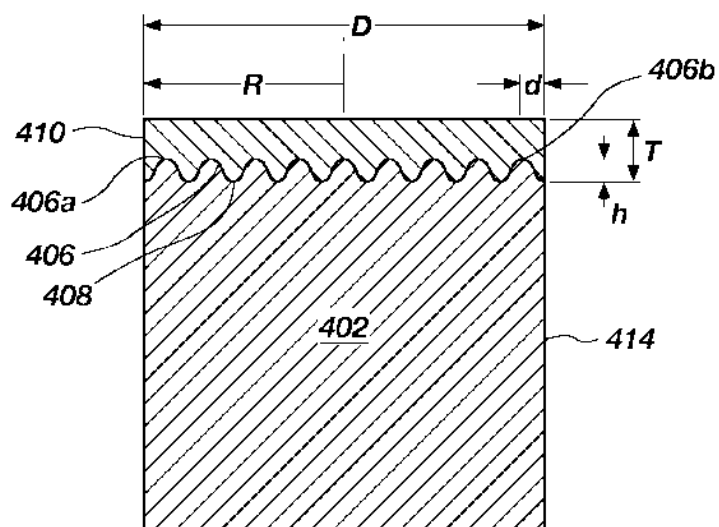


FIG. 4B

U.S. Patent

Dec. 31, 2013

Sheet 7 of 12

US 8,616,306 B2

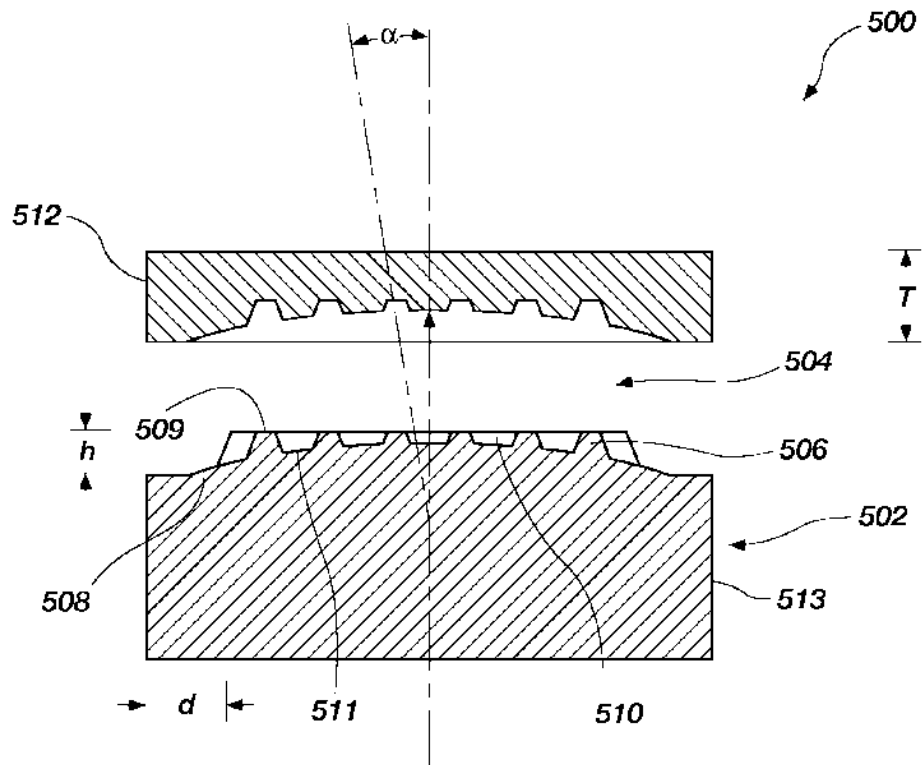


FIG. 5A

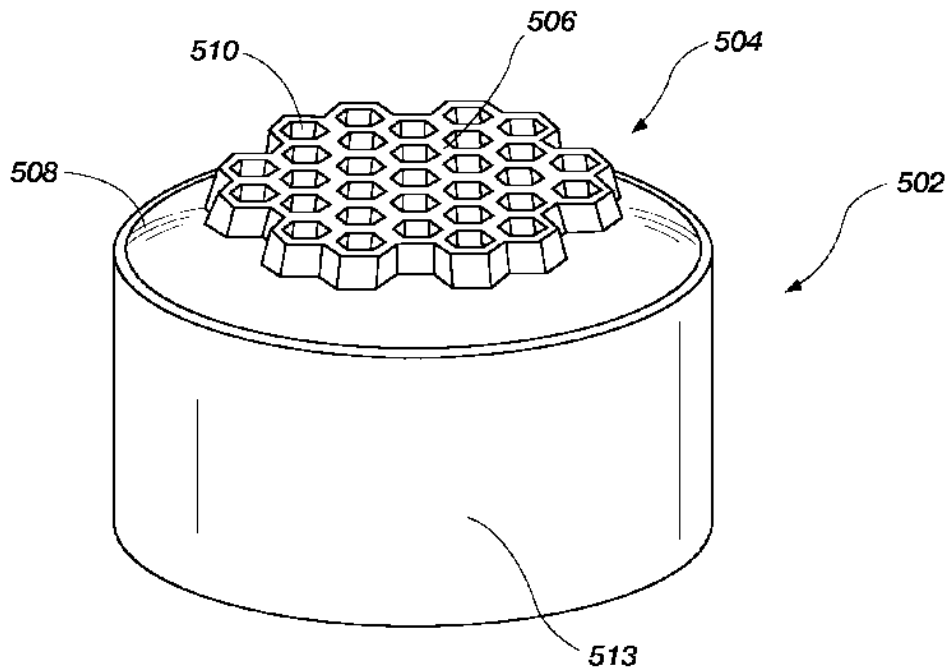


FIG. 5B

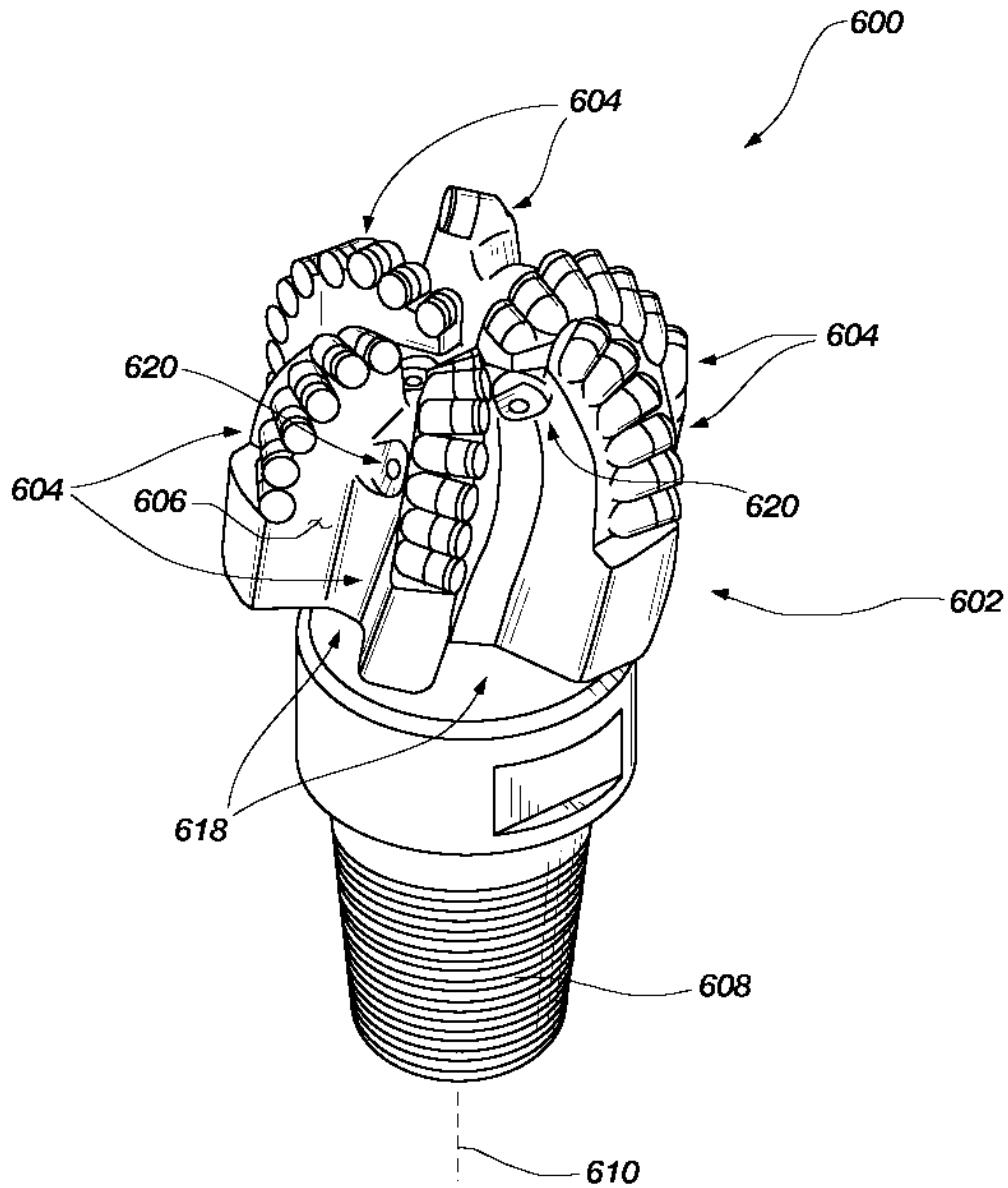


FIG. 6A

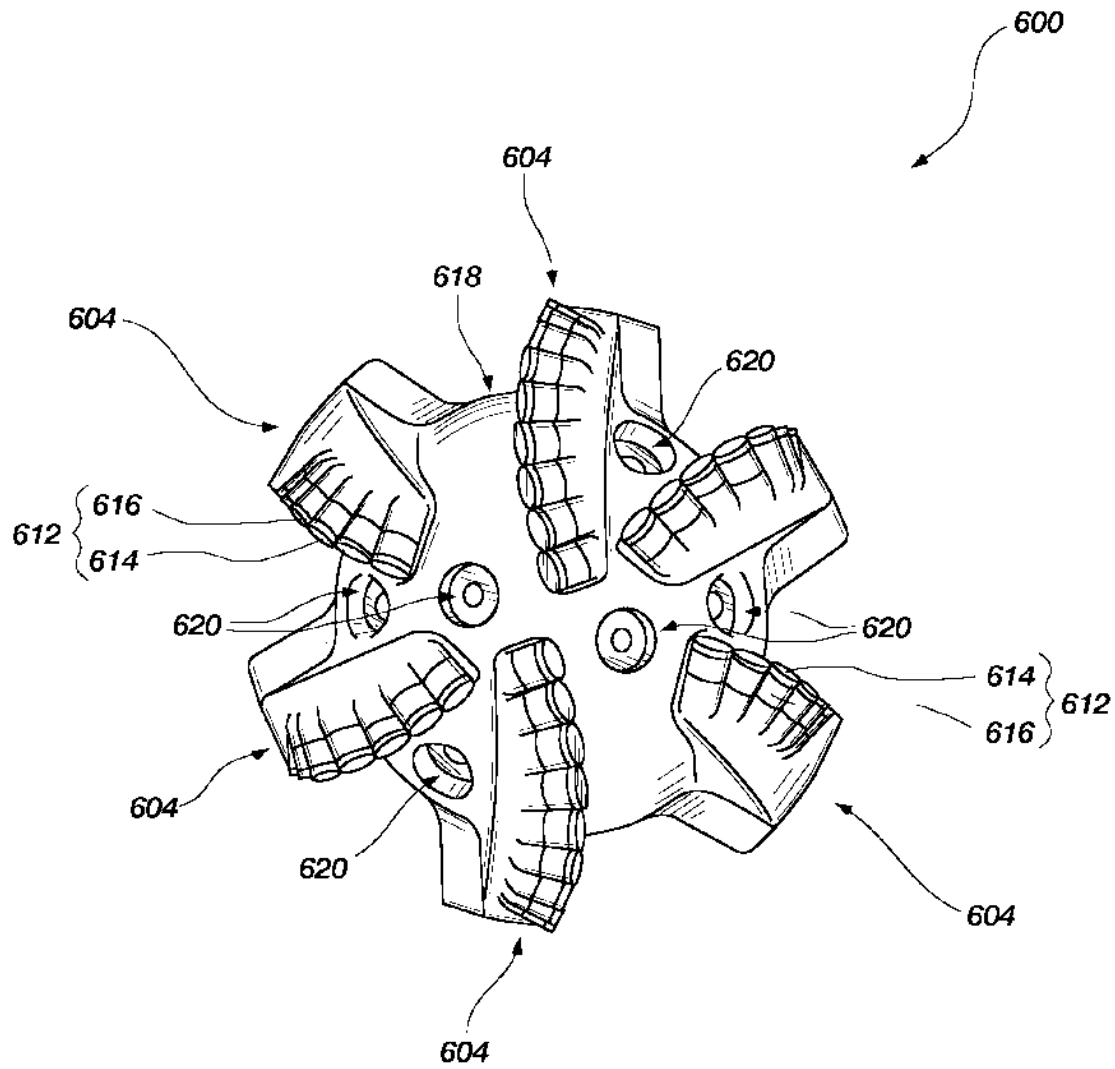


FIG. 6B

U.S. Patent

Dec. 31, 2013

Sheet 10 of 12

US 8,616,306 B2

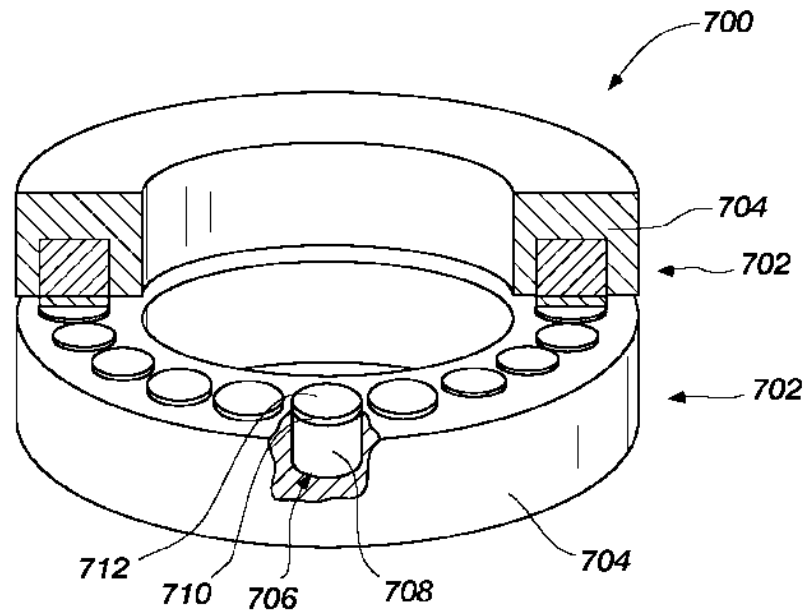


FIG. 7

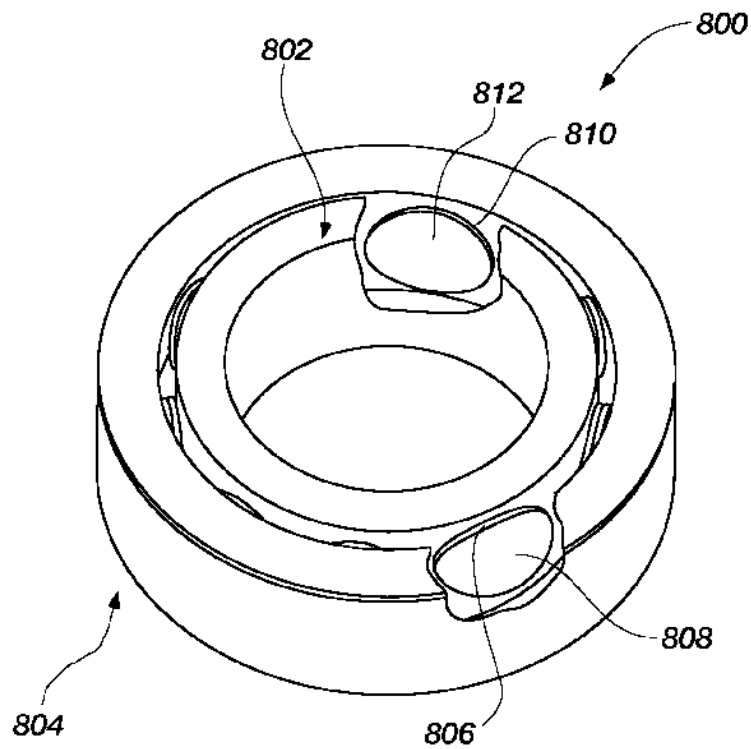


FIG. 8

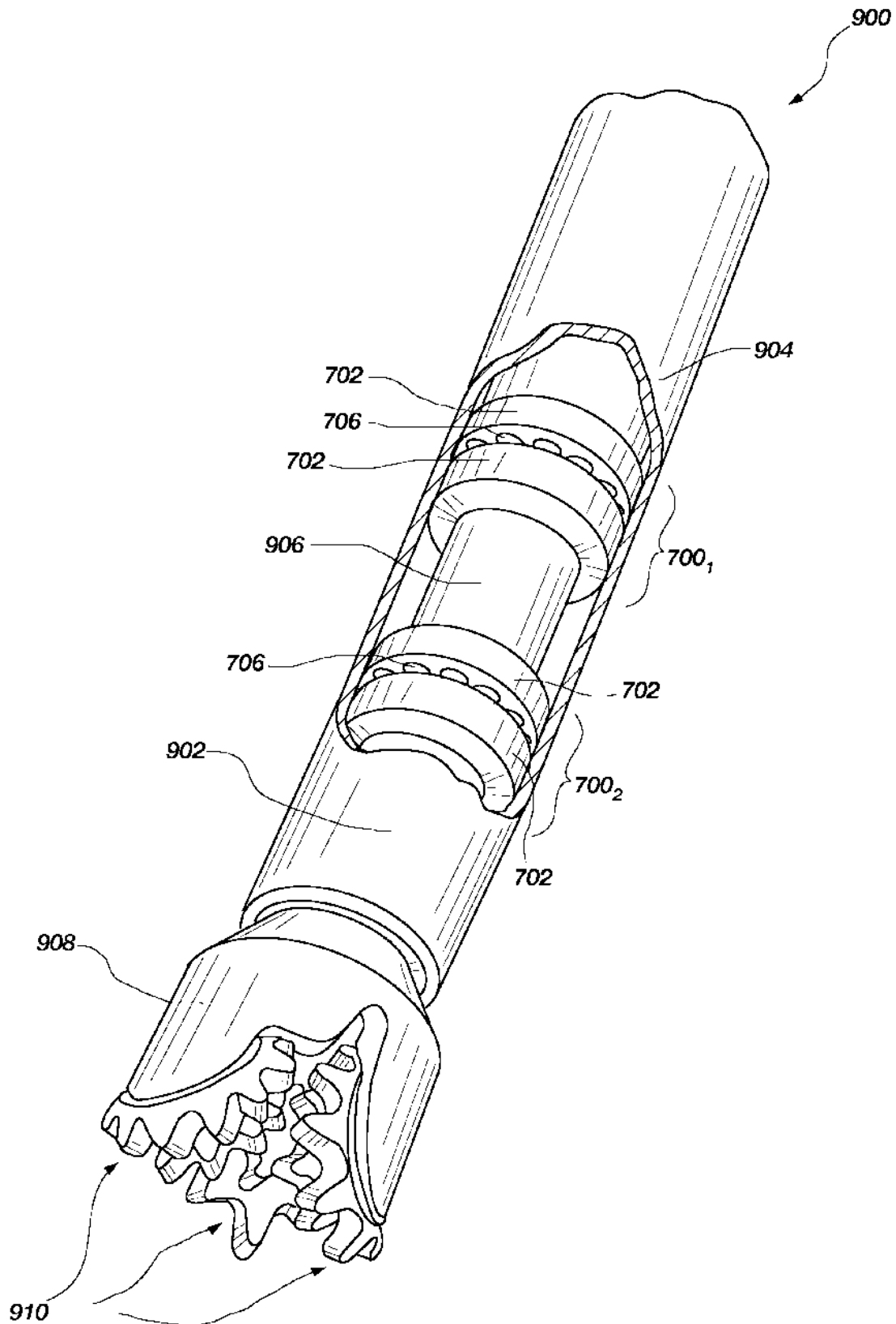


FIG. 9

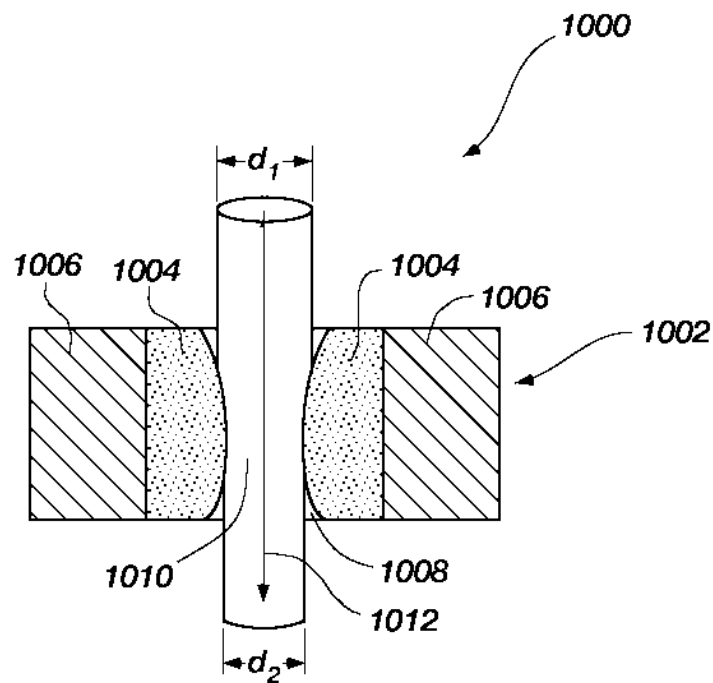


FIG. 10

US 8,616,306 B2

1

POLYCRYSTALLINE DIAMOND COMPACTS, METHOD OF FABRICATING SAME, AND VARIOUS APPLICATIONS

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation of U.S. patent application Ser. No. 12/690,998 filed on 21 Jan. 2010, which is a continuation-in-part of U.S. patent application Ser. No. 12/244,960 filed on 3 Oct. 2008, now U.S. Pat. No. 7,866,418 issued on 11 Jan. 2011, the disclosure of each of which is incorporated herein, in its entirety, by this reference.

BACKGROUND

Wear-resistant, superabrasive compacts are utilized in a variety of mechanical applications. For example, polycrystalline diamond compacts ("PDC's") are used in drilling tools (e.g., cutting elements, gage trimmers, etc.), machining equipment, bearing apparatuses, wire-drawing machinery, and in other mechanical apparatuses.

PDC's have found particular utility as superabrasive cutting elements in rotary drill bits, such as roller cone drill bits and fixed-cutter drill bits. A PDC cutting element typically includes a superabrasive diamond layer commonly referred to as a diamond table. The diamond table may be formed and bonded to a substrate using a high-pressure, high-temperature ("HPHT") process. The PDC cutting element may also be brazed directly into a preformed pocket, socket, or other receptacle formed in a bit body of a rotary drill bit. The substrate may often be brazed or otherwise joined to an attachment member, such as a cylindrical backing. A rotary drill bit typically includes a number of PDC cutting elements affixed to the bit body. A stud carrying the PDC may also be used as a PDC cutting element when mounted to a bit body of a rotary drill bit by press-fitting, brazing, or otherwise securing the stud into a receptacle formed in the bit body.

Conventional PDC's are normally fabricated by placing a cemented carbide substrate into a container with a volume of diamond particles positioned adjacent to the cemented carbide substrate. A number of such cartridges may be loaded into an HPHT press. The substrates and volume of diamond particles are then processed under HPHT conditions in the presence of a catalyst material that causes the diamond particles to bond to one another to form a matrix of bonded diamond grains defining a polycrystalline diamond ("PCD") table that is bonded to the substrate. The catalyst material is often a metal-solvent catalyst (e.g., cobalt, nickel, iron, or alloys thereof) that is used for promoting intergrowth of the diamond particles. For example, a constituent of the cemented carbide substrate, such as cobalt from a cobalt-cemented tungsten carbide substrate, liquefies and sweeps from a region adjacent to the volume of diamond particles into interstitial regions between the diamond particles during the HPHT process. The cobalt acts as a catalyst to promote intergrowth between the diamond particles, which results in formation of bonded diamond grains.

Because of different coefficients of thermal expansion and modulus of elasticity between the PCD table and the cemented carbide substrate, residual stresses of varying magnitudes may develop within different regions of the PCD table and the cemented carbide substrate. Such residual stresses may remain in the PCD table and cemented carbide substrate following cooling and release of pressure from the HPHT process. These complex stresses may be concentrated near the PCD table/substrate interface. Residual stresses at the

2

interface between the PCD table and cemented carbide substrate may result in premature failure of the PDC upon cooling or during subsequent use under thermal stresses and applied forces.

In order to help reduce de-bonding of the PCD table from the cemented carbide substrate, some PDC designers have made the interfacial surface of the cemented carbide substrate that bonds to the PCD table significantly nonplanar. For example, various nonplanar substrate interfacial surface configurations have been proposed and/or used, such as a plurality of spaced protrusions, a honeycomb-type protrusion pattern, and a variety of other configurations.

SUMMARY

Embodiments of the invention relate to PCD exhibiting enhanced diamond-to-diamond bonding. In an embodiment, PCD includes a plurality of diamond grains defining a plurality of interstitial regions. A metal-solvent catalyst occupies at least a portion of the plurality of interstitial regions. The plurality of diamond grains and the metal-solvent catalyst collectively may exhibit a coercivity of about 115 Oersteds ("Oe") or more and a specific magnetic saturation of about 15 Gauss-cm³/grams ("G-cm³/g") or less.

In an embodiment, PCD includes a plurality of diamond grains defining a plurality of interstitial regions. A metal-solvent catalyst occupies the plurality of interstitial regions. The plurality of diamond grains and the metal-solvent catalyst collectively may exhibit a specific magnetic saturation of about 15 G-cm³/g or less. The plurality of diamond grains and the metal-solvent catalyst define a volume of at least about 0.050 cm³.

In an embodiment, a method of fabricating PCD includes enclosing a plurality of diamond particles that exhibit an average particle size of about 30 μ m or less, and a metal-solvent catalyst in a pressure transmitting medium to form a cell assembly. The method further includes subjecting the cell assembly to a temperature of at least about 1000° C., and a pressure in the pressure transmitting medium of at least about 7.5 GPa to form the PCD.

In an embodiment, a PDC includes a PCD table bonded to a substrate. At least a portion of the PCD table may comprise any of the PCD embodiments disclosed herein. In an embodiment, the substrate includes an interfacial surface that is bonded to the polycrystalline diamond table and exhibits a substantially planar topography. According to an embodiment, the interfacial surface may include a plurality of protrusions, and a ratio of a surface area of the interfacial surface in the absence of the plurality of protrusions to a surface area of the interfacial surface with the plurality of protrusions is greater than about 0.600.

In an embodiment, a method of fabricating a PDC includes enclosing a combination in a pressure transmitting medium to form a cell assembly. The combination includes a plurality of diamond particles that exhibit an average particle size of about 30 μ m or less positioned at least proximate to a substrate having an interfacial surface that is substantially planar. The method further includes subjecting the cell assembly to a temperature of at least about 1000° C., and a pressure in the pressure transmitting medium of at least about 7.5 GPa to form a PCD table adjacent to the substrate.

Further embodiments relate to applications utilizing the disclosed PCD and PDC's in various articles and apparatuses, such as rotary drill bits, bearing apparatuses, wire-drawing dies, machining equipment, and other articles and apparatuses.

US 8,616,306 B2

3

Features from any of the disclosed embodiments may be used in combination with one another, without limitation. In addition, other features and advantages of the present disclosure will become apparent to those of ordinary skill in the art through consideration of the following detailed description and the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

The drawings illustrate several embodiments of the invention, wherein identical reference numerals refer to identical elements or features in different views or embodiments shown in the drawings.

FIG. 1A is a schematic diagram of an example of a magnetic saturation apparatus configured to magnetize a PCD sample approximately to saturation.

FIG. 1B is a schematic diagram of an example of a magnetic saturation measurement apparatus configured to measure a saturation magnetization of a PCD sample.

FIG. 2 is a schematic diagram of an example of a coercivity measurement apparatus configured to determine coercivity of a PCD sample.

FIG. 3A is a cross-sectional view of an embodiment of a PDC including a PCD table formed from any of the PCD embodiments disclosed herein.

FIG. 3B is a schematic illustration of a method of fabricating the PDC shown in FIG. 3A according to an embodiment.

FIG. 3C is a graph of residual principal stress versus substrate thickness that was measured in a PCD table of a PDC fabricated at a pressure above about 7.5 GPa and a PCD table of a conventionally formed PDC.

FIG. 4A is an exploded isometric view of a PDC comprising a substrate including an interfacial surface exhibiting a selected substantially planar topography according to an embodiment.

FIG. 4B is an assembled cross-sectional view of the PDC shown in FIG. 4A taken along line 4B-4B.

FIG. 5A is cross-sectional view of a PDC comprising a substrate including an interfacial surface exhibiting a selected substantially planar topography according to yet another embodiment.

FIG. 5B is an isometric view of the substrate shown in FIG. 5A.

FIG. 6A is an isometric view of an embodiment of a rotary drill bit that may employ one or more of the disclosed PDC embodiments.

FIG. 6B is a top elevation view of the rotary drill bit shown in FIG. 6A.

FIG. 7 is an isometric cutaway view of an embodiment of a thrust-bearing apparatus that may utilize one or more of the disclosed PDC embodiments.

FIG. 8 is an isometric cutaway view of an embodiment of a radial bearing apparatus that may utilize one or more of the disclosed PDC embodiments.

FIG. 9 is a schematic isometric cutaway view of an embodiment of a subterranean drilling system including the thrust-bearing apparatus shown in FIG. 7.

FIG. 10 is a side cross-sectional view of an embodiment of a wire-drawing die that employs a PDC fabricated in accordance with the principles described herein.

DETAILED DESCRIPTION

Embodiments of the invention relate to PCD that exhibits enhanced diamond-to-diamond bonding. It is currently believed by the inventors that as the sintering pressure employed during the HPHT process used to fabricate such

4

PCD is moved further into the diamond-stable region away from the graphite-diamond equilibrium line, the rate of nucleation and growth of diamond increases. Such increased nucleation and growth of diamond between diamond particles (for a given diamond particle formulation) may result in PCD being formed exhibiting one or more of a relatively lower metal-solvent catalyst content, a higher coercivity, a lower specific magnetic saturation, or a lower specific permeability (i.e., the ratio of specific magnetic saturation to coercivity) than PCD formed at a lower sintering pressure. Embodiments also relate to PDC's having a PCD table comprising such PCD, methods of fabricating such PCD and PDC's, and applications for such PCD and PDC's in rotary drill bits, bearing apparatuses, wire-drawing dies, machining equipment, and other articles and apparatuses.

PCD Embodiments

According to various embodiments, PCD sintered at a pressure of at least about 7.5 GPa may exhibit a coercivity of 115 Oe or more, a high-degree of diamond-to-diamond bonding, a specific magnetic saturation of about 15 G-cm³/g or less, and a metal-solvent catalyst content of about 7.5 weight % ("wt %") or less. The PCD includes a plurality of diamond grains directly bonded together via diamond-to-diamond bonding (e.g., sp³ bonding) to define a plurality of interstitial regions. At least a portion of the interstitial regions or, in some embodiments, substantially all of the interstitial regions may be occupied by a metal-solvent catalyst, such as iron, nickel, cobalt, or alloys of any of the foregoing metals. For example, the metal-solvent catalyst may be a cobalt-based material including at least 50 wt % cobalt, such as a cobalt alloy.

The diamond grains may exhibit an average grain size of about 50 μm or less, such as about 30 μm or less or about 20 μm or less. For example, the average grain size of the diamond grains may be about 10 μm to about 18 μm and, in some embodiments, about 15 μm to about 18 μm. In some embodiments, the average grain size of the diamond grains may be about 10 μm or less, such as about 2 μm to about 5 μm or submicron. The diamond grain size distribution of the diamond grains may exhibit a single mode, or may be a bimodal or greater grain size distribution.

The metal-solvent catalyst that occupies the interstitial regions may be present in the PCD in an amount of about 7.5 wt % or less. In some embodiments, the metal-solvent catalyst may be present in the PCD in an amount of about 3 wt % to about 7.5 wt %, such as about 3 wt % to about 6 wt %. In other embodiments, the metal-solvent catalyst content may be present in the PCD in an amount less than about 3 wt %, such as about 1 wt % to about 3 wt % or a residual amount to about 1 wt %. By maintaining the metal-solvent catalyst content below about 7.5 wt %, the PCD may exhibit a desirable level of thermal stability suitable for subterranean drilling applications.

Many physical characteristics of the PCD may be determined by measuring certain magnetic properties of the PCD because the metal-solvent catalyst may be ferromagnetic. The amount of the metal-solvent catalyst present in the PCD may be correlated with the measured specific magnetic saturation of the PCD. A relatively larger specific magnetic saturation indicates relatively more metal-solvent catalyst in the PCD.

The mean free path between neighboring diamond grains of the PCD may be correlated with the measured coercivity of the PCD. A relatively large coercivity indicates a relatively smaller mean free path. The mean free path is representative of the average distance between neighboring diamond grains of the PCD, and thus may be indicative of the extent of

US 8,616,306 B2

5

diamond-to-diamond bonding in the PCD. A relatively smaller mean free path, in well-sintered PCD, may indicate relatively more diamond-to-diamond bonding.

As merely one example, ASTM B886-03 (2008) provides a suitable standard for measuring the specific magnetic saturation and ASTM B887-03 (2008) e1 provides a suitable standard for measuring the coercivity of the PCD. Although both ASTM B886-03 (2008) and ASTM B887-03 (2008) e1 are directed to standards for measuring magnetic properties of cemented carbide materials, either standard may be used to determine the magnetic properties of PCD. A KOERZIMAT CS 1.096 instrument (commercially available from Foerster Instruments of Pittsburgh, Pa.) is one suitable instrument that may be used to measure the specific magnetic saturation and the coercivity of the PCD.

Generally, as the sintering pressure that is used to form the PCD increases, the coercivity may increase and the magnetic saturation may decrease. The PCD defined collectively by the bonded diamond grains and the metal-solvent catalyst may exhibit a coercivity of about 115 Oe or more and a metal-solvent catalyst content of less than about 7.5 wt % as indicated by a specific magnetic saturation of about $15 \text{ G}\cdot\text{cm}^3/\text{g}$ or less. In a more detailed embodiment, the coercivity of the PCD may be about 115 Oe to about 250 Oe and the specific magnetic saturation of the PCD may be greater than $0 \text{ G}\cdot\text{cm}^3/\text{g}$ to about $15 \text{ G}\cdot\text{cm}^3/\text{g}$. In an even more detailed embodiment, the coercivity of the PCD may be about 115 Oe to about 175 Oe and the specific magnetic saturation of the PCD may be about $5 \text{ G}\cdot\text{cm}^3/\text{g}$ to about $15 \text{ G}\cdot\text{cm}^3/\text{g}$. In yet an even more detailed embodiment, the coercivity of the PCD may be about 155 Oe to about 175 Oe and the specific magnetic saturation of the PCD may be about $10 \text{ G}\cdot\text{cm}^3/\text{g}$ to about $15 \text{ G}\cdot\text{cm}^3/\text{g}$. The specific permeability (i.e., the ratio of specific magnetic saturation to coercivity) of the PCD may be about 0.10 or less, such as about 0.060 to about 0.090. Despite the average grain size of the bonded diamond grains being less than about $30 \mu\text{m}$ in some embodiments, the metal-solvent catalyst content in the PCD may be less than about 7.5 wt % resulting in a desirable thermal stability.

In one embodiment, diamond particles having an average particle size of about $18 \mu\text{m}$ to about $20 \mu\text{m}$ are positioned adjacent to a cobalt-cemented tungsten carbide substrate and subjected to an HIPIT process at a temperature of about 1390°C . to about 1430°C . and a pressure of about 7.8 GPa to about 8.5 GPa. The PCD so-formed as a PCD table bonded to the substrate may exhibit a coercivity of about 155 Oe to about 175 Oe, a specific magnetic saturation of about $10 \text{ G}\cdot\text{cm}^3/\text{g}$ to about $15 \text{ G}\cdot\text{cm}^3/\text{g}$, and a cobalt content of about 5 wt % to about 7.5 wt %.

In one or more embodiments, a specific magnetic saturation constant for the metal-solvent catalyst in the PCD may be about $185 \text{ G}\cdot\text{cm}^3/\text{g}$ to about $215 \text{ G}\cdot\text{cm}^3/\text{g}$. For example, the specific magnetic saturation constant for the metal-solvent catalyst in the PCD may be about $195 \text{ G}\cdot\text{cm}^3/\text{g}$ to about $205 \text{ G}\cdot\text{cm}^3/\text{g}$. It is noted that the specific magnetic saturation constant for the metal-solvent catalyst in the PCD may be composition dependent.

Generally, as the sintering pressure is increased above 7.5 GPa, a wear resistance of the PCD so-formed may increase. For example, the G_{ratio} may be at least about 4.0×10^6 , such as about 5.0×10^6 to about 15.0×10^6 or, more particularly, about 8.0×10^6 to about 15.0×10^6 . In some embodiments, the G_{ratio} may be at least about 30.0×10^6 . The G_{ratio} is the ratio of the volume of workpiece cut to the volume of PCD worn away during the cutting process. An example of suitable parameters that may be used to determine a G_{ratio} of the PCD are a depth of cut for the PCD cutting element of about 0.254 mm, a back

6

rake angle for the PCD cutting element of about 20 degrees, an in-feed for the PCD cutting element of about 6.35 mm/rev, a rotary speed of the workpiece to be cut of about 101 rpm, and the workpiece may be made from Barre granite having a 914 mm outer diameter and a 254 mm inner diameter. During the G_{ratio} test, the workpiece is cooled with a coolant, such as water.

In addition to the aforementioned G_{ratio} , despite the presence of the metal-solvent catalyst in the PCD, the PCD may exhibit a thermal stability that is close to, substantially the same as, or greater than a partially leached PCD material formed by sintering a substantially similar diamond particle formulation at a lower sintering pressure (e.g., up to about 5.5 GPa) and in which the metal-solvent catalyst (e.g., cobalt) is leached therefrom to a depth of about $60 \mu\text{m}$ to about $100 \mu\text{m}$ from a working surface thereof. The thermal stability of the PCD may be evaluated by measuring the distance cut in a workpiece prior to catastrophic failure, without using coolant, in a vertical lathe test (e.g., vertical turret lathe or a vertical boring mill). An example of suitable parameters that may be used to determine thermal stability of the PCD are a depth of cut for the PCD cutting element of about 1.27 mm, a back rake angle for the PCD cutting element of about 20 degrees, an in-feed for the PCD cutting element of about 1.524 mm/rev, a cutting speed of the workpiece to be cut of about 1.78 msec, and the workpiece may be made from Barre granite having a 914 mm outer diameter and a 254 mm inner diameter. In an embodiment, the distance cut in a workpiece prior to catastrophic failure as measured in the above-described vertical lathe test may be at least about 1300 m, such as about 1300 m to about 3950 m.

PCD formed by sintering diamond particles having the same diamond particle size distribution as a PCD embodiment of the invention, but sintered at a pressure of, for example, up to about 5.5 GPa and at temperatures in which diamond is stable may exhibit a coercivity of about 100 Oe or less and/or a specific magnetic saturation of about $16 \text{ G}\cdot\text{cm}^3/\text{g}$ or more. Thus, in one or more embodiments of the invention, PCD exhibits a metal-solvent catalyst content of less than 7.5 wt % and a greater amount of diamond-to-diamond bonding between diamond grains than that of a PCD sintered at a lower pressure, but with the same precursor diamond particle size distribution and catalyst.

It is currently believed by the inventors that forming the PCD by sintering diamond particles at a pressure of at least about 7.5 GPa may promote nucleation and growth of diamond between the diamond particles being sintered so that the volume of the interstitial regions of the PCD so-formed is decreased compared to the volume of interstitial regions if the same diamond particle distribution was sintered at a pressure of, for example, up to about 5.5 GPa and at temperatures where diamond is stable. For example, the diamond may nucleate and grow from carbon provided by dissolved carbon in metal-solvent catalyst (e.g., liquefied cobalt) infiltrating into the diamond particles being sintered, partially graphitized diamond particles, carbon from a substrate, carbon from another source (e.g., graphite particles and/or fullerenes mixed with the diamond particles), or combinations of the foregoing. This nucleation and growth of diamond in combination with the sintering pressure of at least about 7.5 GPa may contribute to the PCD so-formed having a metal-solvent catalyst content of less than about 7.5 wt %.

FIGS. 1A, 1B, and 2 schematically illustrate the manner in which the specific magnetic saturation and the coercivity of the PCD may be determined using an apparatus, such as the KOERZIMAT CS 1.096 instrument. FIG. 1A is a schematic diagram of an example of a magnetic saturation apparatus 100

US 8,616,306 B2

7

configured to magnetize a PCD sample to saturation. The magnetic saturation apparatus 100 includes a saturation magnet 102 of sufficient strength to magnetize a PCD sample 104 to saturation. The saturation magnet 102 may be a permanent magnet or an electromagnet. In the illustrated embodiment, the saturation magnet 102 is a permanent magnet that defines an air gap 106, and the PCD sample 104 may be positioned on a sample holder 108 within the air gap 106. When the PCD sample 104 is lightweight, it may be secured to the sample holder 108 using, for example, double-sided tape or other adhesive so that the PCD sample 104 does not move responsive to the magnetic field from the saturation magnet 102 and the PCD sample 104 is magnetized at least approximately to saturation.

Referring to the schematic diagram of FIG. 1B, after magnetizing the PCD sample 104 at least approximately to saturation using the magnetic saturation apparatus 100, a magnetic saturation of the PCD sample 104 may be measured using a magnetic saturation measurement apparatus 120. The magnetic saturation measurement apparatus 120 includes a Helmholtz measuring coil 122 defining a passageway dimensioned so that the magnetized PCD sample 104 may be positioned therein on a sample holder 124. Once positioned in the passageway, the sample holder 124 supporting the magnetized PCD sample 104 may be moved axially along an axis direction 126 to induce a current in the Helmholtz measuring coil 122. Measurement electronics 128 are coupled to the Helmholtz measuring coil 122 and configured to calculate the magnetic saturation based upon the measured current passing through the Helmholtz measuring coil 122. The measurement electronics 128 may also be configured to calculate a weight percentage of magnetic material in the PCD sample 104 when the composition and magnetic characteristics of the metal-solvent catalyst in the PCD sample 104 are known, such as with iron, nickel, cobalt, and alloys thereof. Specific magnetic saturation may be calculated based upon the calculated magnetic saturation and the measured weight of the PCD sample 104.

The amount of metal-solvent catalyst in the PCD sample 104 may be determined using a number of different analytical techniques. For example, energy dispersive spectroscopy (e.g., EDAX), wavelength dispersive x-ray spectroscopy (e.g., WDX), Rutherford backscattering spectroscopy, or combinations thereof may be employed to determine the amount of metal-solvent catalyst in the PCD sample 104.

If desired, a specific magnetic saturation constant of the metal-solvent catalyst content in the PCD sample 104 may be determined using an iterative approach. A value for the specific magnetic saturation constant of the metal-solvent catalyst in the PCD sample 104 may be iteratively chosen until a metal-solvent catalyst content calculated by the analysis software of the KOERZIMAT CS 1.09% instrument using the chosen value substantially matches the metal-solvent catalyst content determined via one or more analytical techniques, such as energy dispersive spectroscopy, wavelength dispersive x-ray spectroscopy, or Rutherford backscattering spectroscopy.

FIG. 2 is a schematic diagram of a coercivity measurement apparatus 200 configured to determine a coercivity of a PCD sample. The coercivity measurement apparatus 200 includes a coil 202 and measurement electronics 204 coupled to the coil 202. The measurement electronics 204 are configured to pass a current through the coil 202 so that a magnetic field is generated. A sample holder 206 having a PCD sample 208 thereon may be positioned within the coil 202. A magnetization sensor 210 configured to measure a magnetization of the

8

PCD sample 208 may be coupled to the measurement electronics 204 and positioned in proximity to the PCD sample 208.

During testing, the magnetic field generated by the coil 202 magnetizes the PCD sample 208 at least approximately to saturation. Then, the measurement electronics 204 apply a current so that the magnetic field generated by the coil 202 is increasingly reversed. The magnetization sensor 210 measures a magnetization of the PCD sample 208 resulting from application of the reversed magnetic field to the PCD sample 208. The measurement electronics 204 determine the coercivity of the PCD sample 208, which is a measurement of the strength of the reversed magnetic field at which the magnetization of the PCD sample 208 is zero.

Embodiments of Methods for Fabricating PCD

The PCD may be formed by sintering a mass of a plurality of diamond particles in the presence of a metal-solvent catalyst. The diamond particles may exhibit an average particle size of about 50 μm or less, such as about 30 μm or less, about 20 μm or less, about 10 μm to about 18 μm or, about 15 μm to about 18 μm . In some embodiments, the average particle size of the diamond particles may be about 10 μm or less, such as about 2 μm to about 5 μm or submicron.

In an embodiment, the diamond particles of the mass of diamond particles may comprise a relatively larger size and at least one relatively smaller size. As used herein, the phrases “relatively larger” and “relatively smaller” refer to particle sizes (by any suitable method) that differ by at least a factor of two (e.g., 30 μm and 15 μm). According to various embodiments, the mass of diamond particles may include a portion exhibiting a relatively larger size (e.g., 30 μm , 20 μm , 15 μm , 12 μm , 10 μm , 8 μm) and another portion exhibiting at least one relatively smaller size (e.g., 6 μm , 5 μm , 4 μm , 3 μm , 2 μm , 1 μm , 0.5 μm , less than 0.5 μm , 0.1 μm , less than 0.1 μm). In one embodiment, the mass of diamond particles may include a portion exhibiting a relatively larger size between about 10 μm and about 40 μm and another portion exhibiting a relatively smaller size between about 1 μm and 4 μm . In some embodiments, the mass of diamond particles may comprise three or more different sizes (e.g., one relatively larger size and two or more relatively smaller sizes), without limitation.

It is noted that the as-sintered diamond grain size may differ from the average particle size of the mass of diamond particles prior to sintering due to a variety of different physical processes, such as grain growth, diamond particle fracturing, carbon provided from another carbon source (e.g., dissolved carbon in the metal-solvent catalyst), or combinations of the foregoing. The metal-solvent catalyst (e.g., iron, nickel, cobalt, or alloys thereof) may be provided in particulate form mixed with the diamond particles, as a thin foil or plate placed adjacent to the mass of diamond particles, from a cemented carbide substrate including a metal-solvent catalyst, or combinations of the foregoing.

In order to efficiently sinter the mass of diamond particles, the mass may be enclosed in a pressure transmitting medium, such as a refractory metal can, graphite structure, pyrophyllite, combinations thereof, or other suitable pressure transmitting structure to form a cell assembly. Examples of suitable gasket materials and cell structures for use in manufacturing PCD are disclosed in U.S. Pat. No. 6,338,754 and U.S. patent application Ser. No. 11/545,929, each of which is incorporated herein, in its entirety, by this reference. Another example of a suitable pressure transmitting material is pyrophyllite, which is commercially available from Wonderstone Ltd. of South Africa. The cell assembly, including

US 8,616,306 B2

9

the pressure transmitting medium and mass of diamond particles therein, is subjected to an HPHT process using an ultra-high pressure press at a temperature of at least about 1000° C. (e.g., about 1100° C. to about 2200° C., or about 1200° C. to about 1450° C.) and a pressure in the pressure transmitting medium of at least about 7.5 GPa (e.g., about 7.5 GPa to about 15 GPa, about 9 GPa to about 12 GPa, or about 10 GPa to about 12.5 GPa) for a time sufficient to sinter the diamond particles together in the presence of the metal-solvent catalyst and form the PCD comprising bonded diamond grains defining interstitial regions occupied by the metal-solvent catalyst. For example, the pressure in the pressure transmitting medium employed in the HPHT process may be at least about 8.0 GPa, at least about 9.0 GPa, at least about 10.0 GPa, at least about 11.0 GPa, at least about 12.0 GPa, or at least about 14 GPa.

The pressure values employed in the HPHT processes disclosed herein refer to the pressure in the pressure transmitting medium at room temperature (e.g., about 25° C.) with application of pressure using an ultra-high pressure press and not the pressure applied to exterior of the cell assembly. The actual pressure in the pressure transmitting medium at sintering temperature may be slightly higher. The ultra-high pressure press may be calibrated at room temperature by embedding at least one calibration material that changes structure at a known pressure such as, PbTe, thallium, barium, or bismuth in the pressure transmitting medium. Optionally, a change in resistance may be measured across the at least one calibration material due to a phase change thereof. For example, PbTe exhibits a phase change at room temperature at about 6.0 GPa and bismuth exhibits a phase change at room temperature at about 7.7 GPa. Examples of suitable pressure calibration techniques are disclosed in G. Rousse, S. Klotz, A. M. Saitta, J. Rodriguez-Carvajal, M. J. McMahon, B. Couzinet, and M. Mezouar, "Structure of the Intermediate Phase of PbTe at High Pressure," *Physical Review B: Condensed Matter and Materials Physics*, 71, 224116 (2005) and D. L. Decker, W. A. Bassett, L. Merrill, H. T. Hall, and J. D. Barnett, "High-Pressure Calibration: A Critical Review," *J. Phys. Chem. Ref. Data*, 1, 3 (1972).

In an embodiment, a pressure of at least about 7.5 GPa in the pressure transmitting medium may be generated by applying pressure to a cubic high-pressure cell assembly that encloses the mass of diamond particles to be sintered using anvils, with each anvil applying pressure to a different face of the cubic high-pressure assembly. In such an embodiment, a surface area of each anvil face of the anvils may be selectively dimensioned to facilitate application of pressure of at least about 7.5 GPa to the mass of diamond particles being sintered. For example, the surface area of each anvil may be less than about 16.0 cm², such as less than about 16.0 cm², about 8 cm² to about 10 cm². The anvils may be made from a cobalt-cemented tungsten carbide or other material having a sufficient compressive strength to help reduce damage thereto through repetitive use in a high-volume commercial manufacturing environment. As an alternative to or in addition to selectively dimensioning the surface area of each anvil face, in an embodiment, two or more internal anvils may be embedded in the cubic high-pressure cell assembly to further intensify pressure. For example, the article W. Utsumi, N. Toyama, S. Endo and F. I. Fujita, "X-ray diffraction under ultrahigh pressure generated with sintered diamond anvils," *J. Appl. Phys.*, 60, 2201 (1986) is incorporated herein, in its entirety, by this reference and discloses that sintered diamond anvils may be embedded in a cubic pressure transmitting medium

10

for intensifying the pressure applied by an ultra-high pressure press to a workpiece also embedded in the cubic pressure transmitting medium.

PIXC Embodiments and Methods of Fabricating PIXCs

Referring to FIG. 3A, the PCD embodiments may be employed in a PIXC for cutting applications, bearing applications, or many other applications. FIG. 3A is a cross-sectional view of an embodiment of a PIXC 300. The PIXC 300 includes a substrate 302 bonded to a PCD table 304. The PCD table 304 may be formed of PCD in accordance with any of the PCD embodiments disclosed herein. The PCD table 304 exhibits at least one working surface 306 and at least one lateral dimension "L" (e.g., a diameter). Although FIG. 3A shows the working surface 306 as substantially planar, the working surface 306 may be concave, convex, or another nonplanar geometry. Furthermore, other regions of the PCD table 304 may function as a working region, such as a peripheral side surface and/or an edge. The substrate 302 may be generally cylindrical or another selected configuration, without limitation. Although FIG. 3A shows an interfacial surface 308 of the substrate 302 as being substantially planar, the interfacial surface 308 may exhibit a selected nonplanar topography, such as a grooved, ridged, or other nonplanar interfacial surface. The substrate 302 may include, without limitation, cemented carbides, such as tungsten carbide, titanium carbide, chromium carbide, niobium carbide, tantalum carbide, vanadium carbide, or combinations thereof cemented with iron, nickel, cobalt, or alloys thereof. For example, in one embodiment, the substrate 302 comprises cobalt-cemented tungsten carbide.

In some embodiments, the PCD table 304 may include two or more layered regions 310 and 312 exhibiting different compositions and/or different average diamond grain sizes. For example, the region 310 is located adjacent to the interface surface 308 of the substrate 302 and exhibits a first diamond grain size, while the region 312 is remote from the substrate 302 and exhibits a second average diamond grain size that is less than that of the first average diamond grain size. For example, the second average diamond grain size may be about 90% to about 98% (e.g., about 90 to about 95%) of the first diamond grain size. In another embodiment, the second average diamond grain size may be greater than that of the first average diamond grain size. For example, the first average diamond grain size may be about 90% to about 98% (e.g., about 90 to about 95%) of the second diamond grain size.

As an alternative to or in addition to the first and second regions exhibiting different diamond grain sizes, in an embodiment, the composition of the region 310 may be different than that of the region 312. The region 310 may include about 15 wt % or less of a tungsten-containing material (e.g., tungsten and/or tungsten carbide) interspersed between the diamond grains to improve toughness, while the region 312 may be substantially free of tungsten. For example, the tungsten-containing material may be present in the region 310 in an amount of about 1 wt % to about 10 wt %, about 5 wt % to about 10 wt %, or about 10 wt %.

FIG. 3B is a schematic illustration of an embodiment of a method for fabricating the PIXC 300 shown in FIG. 3A. Referring to FIG. 3B, a mass of diamond particles 305 having any of the above-mentioned average particle sizes and distributions (e.g., an average particle size of about 50 μm or less) is positioned adjacent to the interfacial surface 308 of the substrate 302. As previously discussed, the substrate 302 may

US 8,616,306 B2

11

include a metal-solvent catalyst. The mass of diamond particles **305** and substrate **302** may be subjected to an HPHT process using any of the conditions previously described with respect to sintering the PCD embodiments disclosed herein. The PDC **300** so-formed includes the PCD table **304** that comprises PCD, formed of any of the PCD embodiments disclosed herein, integrally formed with the substrate **302** and bonded to the interfacial surface **308** of the substrate **302**. If the substrate **302** includes a metal-solvent catalyst, the metal-solvent catalyst may liquefy and infiltrate the mass of diamond particles **305** to promote growth between adjacent diamond particles of the mass of diamond particles **305** to form the PCD table **304** comprised of a body of bonded diamond grains having the infiltrated metal-solvent catalyst interstitially disposed between bonded diamond grains. For example, if the substrate **302** is a cobalt-cemented tungsten carbide substrate, cobalt from the substrate **302** may be liquefied and infiltrate the mass of diamond particles **305** to catalyze formation of the PCD table **304**.

In some embodiments, the mass of diamond particles **305** may include two or more layers exhibiting different compositions and/or different average diamond particle sizes. For example, a first layer may be located adjacent to the interface surface **308** of the substrate **302** and exhibit a first diamond particle size, while a second layer may be located remote from the substrate **302** and exhibit a second average diamond particle size that is less than that of the first average diamond particle size. For example, the second average diamond particle size may be about 90% to about 98% (e.g., about 90 to about 95%) of the first diamond particle size. In another embodiment, the second average diamond particle size may be greater than that of the first average diamond particle size. For example, the first average diamond particle size may be about 90% to about 98% (e.g., about 90 to about 95%) of the second diamond particle size.

As an alternative to or in addition to the first and second layers exhibiting different diamond particles sizes, in an embodiment, the composition of the first layer may be different than that of the second layer. The first layer may include about 15 wt % or less of a tungsten-containing material (e.g., tungsten and/or tungsten carbide) mixed with the diamond particles, while the second layer may be substantially free of tungsten. For example, the tungsten-containing material may be present in the first layer in an amount of about 1 wt % to about 10 wt %, about 5 wt % to about 10 wt %, or about 10 wt %.

Employing selectively dimensioned anvil faces and/or internal anvils in the ultra-high pressure press used to process the mass of diamond particles **305** and substrate **302** enables forming the at least one lateral dimension *d* of the PCD table **304** to be about 0.80 cm or more. Referring again to FIG. 3A, for example, the at least one lateral dimension "*D*" may be about 0.80 cm to about 3.0 cm and, in some embodiments, about 1.3 cm to about 1.9 cm or about 1.6 cm to about 1.9 cm. A representative volume of the PCD table **304** (or any PCD article of manufacture disclosed herein) formed using the selectively dimensioned anvil faces and/or internal anvils may be at least about 0.050 cm³. For example, the volume may be about 0.25 cm³ to at least about 1.25 cm³ or about 0.1 cm³ to at least about 0.70 cm³. A representative volume for the PDC **300** may be about 0.4 cm³ to at least about 4.6 cm³, such as about 1.1 cm³ to at least about 2.3 cm³.

In other embodiments, a PCD table according to an embodiment may be separately formed using an HPHT sintering process (i.e., a pre-sintered PCD table) and, subsequently, bonded to the interfacial surface **308** of the substrate **302** by brazing, using a separate HPHT bonding process, or

12

any other suitable joining technique, without limitation. In yet another embodiment, a substrate may be formed by depositing a binderless carbide (e.g., tungsten carbide) via chemical vapor deposition onto the separately formed PCD table.

In any of the embodiments disclosed herein, substantially all or a selected portion of the metal-solvent catalyst may be removed (e.g., via leaching) from the PCD table. In an embodiment, metal-solvent catalyst in the PCD table may be removed to a selected depth from at least one exterior working surface (e.g., the working surface **306** and/or a sidewall working surface of the PCD table **304**) so that only a portion of the interstitial regions are occupied by metal-solvent catalyst. For example, substantially all or a selected portion of the metal-solvent catalyst may be removed from the PCD table **304** of the PDC **300** to a selected depth from the working surface **306**.

In another embodiment, a PCD table may be fabricated according to any of the disclosed embodiments in a first HPHT process, leached to remove substantially all of the metal-solvent catalyst from the interstitial regions between the bonded diamond grains, and subsequently bonded to a substrate in a second HPHT process. In the second HPHT process, an infiltrant from, for example, a cemented carbide substrate may infiltrate into the interstitial regions from which the metal-solvent catalyst was depleted. For example, the infiltrant may be cobalt that is swept-in from a cobalt-cemented tungsten carbide substrate. In one embodiment, the first and/or second HPHT process may be performed at a pressure of at least about 7.5 GPa. In one embodiment, the infiltrant may be leached from the infiltrated PCD table using a second acid leaching process following the second HPHT process.

In some embodiments, the pressure employed in the HPHT process used to fabricate the PDC **300** may be sufficient to reduce residual stresses in the PCD table **304** that develop during the HPHT process due to the thermal expansion mismatch between the substrate **302** and the PCD table **304**. In such an embodiment, the principal stress measured on the working surface **306** of the PDC **300** may exhibit a value of about -345 MPa to about 0 MPa, such as about -289 MPa. For example, the principal stress measured on the working surface **306** may exhibit a value of about -345 MPa to about 0 MPa. A conventional PDC fabricated using an HPHT process at a pressure below about 7.5 GPa may result in a PCD table thereof exhibiting a principal stress on a working surface thereof of about -1724 MPa to about -414 MPa, such as about -770 MPa.

Residual stress may be measured on the working surface **306** of the PCD table **304** of the PDC **300** as described in T. P. Lin, M. Hood, G. A. Cooper, and R. H. Smith, "Residual stresses in polycrystalline diamond compacts," J. Am. Ceram. Soc. 77, 6, 1562-1568 (1994). More particularly, residual strain may be measured with a rosette strain gage bonded to the working surface **306**. Such strain may be measured for different levels of removal of the substrate **302** (e.g., as material is removed from the back of the substrate **302**). Residual stress may be calculated from the measured residual strain data.

FIG. 3C is a graph of residual principal stress versus substrate thickness that was measured in a PCD table of a PDC fabricated at pressure above about 7.5 GPa in accordance with an embodiment of the invention and a PCD table of a conventionally formed PDC. The substrate of each PDC had a substantially planar interfacial surface. The residual principal stress was determined using the technique described in the article referenced above by Lin et al. Curve **310** shows the

US 8,616,306 B2

13

measured residual principal stress on a working surface of the PDC fabricated at a pressure above about 7.5 GPa. The PDC that was fabricated at a pressure above about 7.5 GPa had a PCD table thickness dimension of about 1 mm and the substrate had a thickness dimension of about 7 mm and a diameter of about 13 mm. Curve 312 shows the measured residual principal stress on a working surface of a PCD table of a conventionally PDC fabricated at pressure below about 7.5 GPa. The PDC that was fabricated at a pressure below about 7.5 GPa had a PCD table thickness dimension of about 1 mm and the substrate had a thickness dimension of about 7 mm and a diameter of about 13 mm. The highest absolute value of the residual principal stress occurs with the full substrate length of about 7 mm. As shown by the curves 310 and 312, increasing the pressure employed in the HIPIT process used to fabricate a PDC, above about 7.5 GPa may reduce the highest absolute value of the principal residual stress in a PCD table thereof by about 60% relative to a conventionally fabricated PDC. For example, at the full substrate length, the absolute value of the principal residual stress in the PCD table fabricated at a pressure above about 7.5 GPa is about 60% less than the absolute value of the principal residual stress in the PCD table of the conventionally fabricated PDC.

As discussed above in relation to FIG. 3C, the application of higher pressure in the HIPIT process used to fabricate a PDC may substantially reduce the residual compressive stresses in the PCD table. Typically, high residual compressive stresses in the PCD table are believed desirable to help reduce crack propagation in the PCD table. The inventors have found that the reduced residual compressive stresses in a PCD table of a PDC fabricated in an HIPIT process at a pressure of at least about 7.5 GPa may result in detrimental cracking in the PCD table and de-bonding of the PCD table from the substrate upon brazing the substrate to, for example, a carbide extension and/or a bit body of a rotary drill bit depending upon the extent of the nonplanarity of the interfacial surface of the substrate. It is believed by the inventors that when the PDC is fabricated at a pressure of at least about 7.5 GPa, at the brazing temperature, tensile stresses generated in the PCD table due to thermal expansion are greater than if the PCD table had higher residual compressive stresses. Due to the higher tensile stresses at the brazing temperature, hoop stresses generated in the PCD by nonplanar surface features (e.g., protrusions) of the substrate may cause the PCD table to form radially-extending and vertically-extending cracks and/or de-bond from the substrate more frequently than if fabricated at relatively lower pressures. Typically, conventional wisdom taught that a highly nonplanar interfacial surface for the substrate helped prevent de-bonding of the PCD table from the substrate. Thus, in certain embodiments discussed in more detail in FIGS. 3A-6B, the inventors have proceeded contrary to conventional wisdom, which suggested that a highly nonplanar interfacial surface for the substrate promotes bonding. In such embodiments, the topography of the interfacial surface of the substrate may be controlled so that it is still substantially planar and exhibits a nonplanarity that does not exceed a maximum threshold.

Referring again to FIG. 3A, in an embodiment, the interfacial surface 308 of the substrate 302 may be substantially planar. For example, to the extent that the interfacial surface 308 includes a plurality of protrusions, the protrusions may exhibit an average surface relief height of about 0 to less than about 0.00010 inch, about 0 to about 0.00050 inch, about 0 to about 0.00075 inch, or about 0.000010 inch to about 0.00010 inch. The average surface relief is the height that the protrusions extend above the lowest point of the interfacial surface 308. A ratio of a surface area of the interfacial surface in the

14

absence of the plurality of protrusions (i.e., a flat interfacial surface) to a surface area of the interfacial surface with the plurality of protrusions is greater than about 0.600. An example of an interfacial surface that is substantially planar is one in which the ratio is greater than about 0.600. For example, the ratio may be about 0.600 to about 0.650, about 0.650 to about 0.725, about 0.650 to about 0.750, about 0.650 to about 0.950, about 0.750 to less than 1.0, or about 0.750 to about 1.0.

FIGS. 4A-6B illustrate embodiments in which the selected substantially planar topography of the interfacial surface of the substrate is controlled to reduce or substantially eliminate cracking in and/or de-bonding of a PCD table of a PDC. FIGS. 4A and 4B are exploded isometric and assembled isometric views, respectively, of an embodiment of a PDC 400 comprising a substrate 402 including an interfacial surface 404 exhibiting a selected substantially planar topography. The substrate 402 may be made from the same carbide materials as the substrate 302 shown in FIG. 3A. The interfacial surface 404 includes a plurality of protrusions 406 spaced from each other and extending substantially transversely to the length of the substrate 402. The protrusions 406 define a plurality of grooves 408 between pairs of the protrusions 406. A PCD table 410 may be bonded to the interfacial surface 406. The PCD table 410 may exhibit some or all of the magnetic, mechanical, thermal stability, wear resistance, size, compositional, diamond-to-diamond bonding, or grain size properties of the PCD disclosed herein and/or the PCD table 304 shown in FIG. 3A. The PCD table 410 exhibits a maximum thickness "T." Because the PCD table 410 may be integrally formed with the substrate 402 and fabricated from precursor diamond particles, the PCD table 410 may have an interfacial surface 411 that is configured to correspond to the topography of the interfacial surface 404 of the substrate 402.

A ratio of a surface area of the interfacial surface 404 in the absence of the plurality of protrusions 406 (i.e., a flat interfacial surface) to a surface area of the interfacial surface with the protrusions 406 is greater than about 0.600. For example, the ratio may be about 0.600 to about 0.650, about 0.650 to about 0.725, about 0.650 to about 0.750, about 0.650 to about 0.950, about 0.750 to less than 1.0, or about 0.750 to about 1.0.

The plurality of protrusions 406 exhibits an average surface relief height "h," which is the average height that the protrusions 406 extend above the lowest point of the interfacial surface 404. For example, h may be greater than 0 to less than about 0.030 inch, greater than 0 to about 0.020 inch, greater than 0 to about 0.015 inch, about 0.0050 inch to about 0.010 inch, or 0.0080 inch to about 0.010 inch. The maximum thickness "T" may be about 0.050 inch to about 0.20 inch, such as about 0.050 inch to about 0.16 inch, about 0.050 inch to about 0.10 inch, about 0.050 inch to about 0.085 inch or about 0.070 inch to about 0.080 inch. The ratio of h/T may be less than about 0.25, such as about 0.050 to about 0.125, about 0.050 to about 0.10, about 0.070 to about 0.090, or about 0.050 to about 0.075.

Referring to FIG. 4B, the outermost of the protrusions 406 (indicated as 406a and 406b) may be laterally spaced from an exterior peripheral surface 414 of the substrate 402 by a distance d. When the PDC 400 is substantially cylindrical, a ratio of d to the radius of the PCD table "R" may be about 0.030 to about 1.0, about 0.035 to about 0.080, or about 0.038 to about 0.060.

FIG. 5A is cross-sectional view of a PDC 500 comprising a substrate 502 including an interfacial surface 504 exhibiting a selected substantially planar topography according to yet another embodiment and FIG. 5B is an isometric view of the

US 8,616,306 B2

15

substrate 502. The substrate 502 may be made from the same carbide materials as the substrate 302 shown in FIG. 3A. The interfacial surface 504 of the substrate 502 includes a plurality of hexagonal protrusions 506 that extend outwardly from a face 508. The face 508 may be convex, as in the illustrated embodiment, or substantially planar. Tops 509 of the protrusions 506 may lie generally in a common plane. The plurality of protrusions 506 defines a plurality of internal cavities 510. A depth of each internal cavity 510 may decrease as they approach the center of the substrate 502. A bottom 511 of each cavity 510 may follow the profile of the face 508.

The PDC 500 further includes a PCD table 512 exhibiting a maximum thickness "T," which is bonded to the interfacial surface 504 of the substrate 502. The thickness of the PCD table 512 gradually increases with lateral distance from the center of the PCD table 512 toward a perimeter 513 of the PDC 500. The PCD table 512 may be configured to correspond to the topography of the interfacial surface 504 of the substrate 502. For example, protrusions 513 of the PCD table 512 may fill each of the internal cavities 510 defined by the protrusions 506 of the substrate 502. The PCD table 512 may exhibit some or all of the magnetic, mechanical, thermal stability, wear resistance, size, compositional, diamond-to-diamond bonding, or grain size properties of the PCD disclosed herein and/or the PCD table 304 shown in FIG. 3A. The closed features of the hexagonal protrusions 506 include a draft angle α , such as about 5 degrees to about 15 degrees.

A ratio of a surface area of the interfacial surface 504 in the absence of the protrusions 506 (i.e., a flat interfacial surface) to a surface area of the interfacial surface with the protrusions 506 is greater than about 0.600. For example, the ratio may be about 0.600 to about 0.650, about 0.650 to about 0.725, about 0.650 to about 0.750, about 0.650 to about 0.950, about 0.750 to less than 1.0, or about 0.750 to about 1.0.

The plurality of protrusions 506 exhibits an average surface relief height "h," which is the average height that the protrusions 506 extend above the lowest point of the interfacial surface 504. For example, h may be greater than 0 to less than about 0.030 inch, greater than 0 to about 0.020 inch, greater than 0 to about 0.015 inch, about 0.0050 inch to about 0.010 inch, or 0.0080 inch to about 0.010 inch. The maximum thickness "T" may be about 0.050 inch to about 0.10 inch, such as about 0.050 inch to about 0.085 inch or about 0.070 inch to about 0.080 inch. The ratio of h/T may be less than about 0.25, such as about 0.050 to about 0.125, about 0.050 to about 0.10, about 0.070 to about 0.090, or about 0.050 to about 0.075.

It is noted that the interfacial surface geometries shown in the PDCs 400 and 500 are merely two examples of suitable interfacial surface geometries. Other interfacial surface

16

geometries may be employed that depart from the illustrated interfacial surface geometries shown in the PDCs 400 and 500 of FIGS. 4A-5B.

Working Examples

The following working examples provide further detail about the magnetic properties of PCD tables of PDCs fabricated in accordance with the principles of some of the specific embodiments of the invention. The magnetic properties of each PCD table listed in Tables I-IV were tested using a KOERZIMAT CS 1.096 instrument that is commercially available from Foerster Instruments of Pittsburgh, Pa. The specific magnetic saturation of each PCD table was measured in accordance with ASTM B886-03 (2008) and the coercivity of each PCD table was measured using ASTM B887-03 (2008) e1 using a KOERZIMAT CS 1.096 instrument. The amount of cobalt-based metal-solvent catalyst in the tested PCD tables was determined using energy dispersive spectroscopy and Rutherford backscattering spectroscopy. The specific magnetic saturation constant of the cobalt-based metal-solvent catalyst in the tested PCD tables was determined to be about 201 G \cdot cm³/g using an iterative analysis as previously described. When a value of 201 G \cdot cm³/g was used for the specific magnetic saturation constant of the cobalt-based metal-solvent catalyst, the calculated amount of the cobalt-based metal-solvent catalyst in the tested PCD tables using the analysis software of the KOERZIMAT CS 1.096 instrument substantially matched the measurements using energy dispersive spectroscopy and Rutherford spectroscopy.

Table 1 below lists PCD tables that were fabricated in accordance with the principles of certain embodiments of the invention discussed above. Each PCD table was fabricated by placing a mass of diamond particles having the listed average diamond particle size adjacent to a cobalt-cemented tungsten carbide substrate in a niobium container, placing the container in a high-pressure cell medium, and subjecting the high-pressure cell medium and the container therein to an HPHT process using an HPHT cubic press to form a PCD table bonded to the substrate. The surface area of each anvil of the HPHT press and the hydraulic line pressure used to drive the anvils were selected so that the sintering pressure was at least about 7.8 GPa. The temperature of the HPHT process was about 1400° C, and the sintering pressure was at least about 7.8 GPa. The sintering pressures listed in Table 1 refer to the pressure in the high-pressure cell medium at room temperature, and the actual sintering pressures at the sintering temperature are believed to be greater. After the HPHT process, the PCD table was removed from the substrate by grinding away the substrate. However, the substrate may also be removed using electro-discharge machining or another suitable method.

TABLE I

Example	Average Diamond Particle Size (um)	Sintering Pressure (GPa)	Specific Magnetic Saturation (G \cdot cm ³ /g)	Calculated Co wt %	Coercivity (Oe)	Specific Permeability (G \cdot cm ³ /g \cdot Oe)
1	20	7.8	11.15	5.549	150.2	0.008564
2	19	7.8	11.64	5.797	170.0	0.008847
3	19	7.8	11.85	5.899	157.9	0.008505
4	19	7.8	11.15	5.550	170.9	0.008524
5	19	7.8	11.43	5.689	163.5	0.008987
6	19	7.8	10.67	5.120	145.9	0.007263
7	19	7.8	10.75	5.357	152.3	0.007065

US 8,616,306 B2

17

TABLE I-continued

Selected Magnetic Properties of PCD Tables Fabricated According to Impediments of the Invention						
Example	Average Diamond Particle Size (μm)	Sintering Pressure (GPa)	Specific Magnetic Saturation ($\text{G} \cdot \text{cm}^3/\text{g}$)	Calculated Co wt %	Coercivity (Oe)	Specific Permeability ($\text{G} \cdot \text{cm}^3/\text{g} \cdot \text{Oe}$)
8	19	7.8	10.22	5.087	145.2	0.07039
9	19	7.8	10.12	5.041	156.6	0.06469
10	19	7.8	10.72	5.249	137.1	0.07819
11	11	7.8	12.52	6.329	135.3	0.09254
12	11	7.8	12.78	6.369	130.5	0.09793
13	11	7.8	12.69	6.315	134.6	0.09428
14	11	7.8	13.20	6.569	131.6	0.1003

Table II below lists conventional PCD tables that were fabricated. Each PCD table listed in Table II was fabricated by placing a mass of diamond particles having the listed average diamond particle size adjacent to a cobalt-cemented tungsten carbide substrate in a niobium container, placing container in a high-pressure cell medium, and subjecting the high-pressure cell medium and the container therein to an HPHT process using an HPHT cubic press to form a PCD table bonded to the substrate. The surface area of each anvil of the HPHT press and the hydraulic line pressure used to drive the anvils were selected so that the sintering pressure was about 4.6 GPa. Except for samples 15, 16, 18, and 19, which were subjected to a temperature of about 1430° C., the temperature of the HPHT process was about 1400° C., and the sintering pressure was about 4.6 GPa. The sintering pressures listed in Table II refer to the pressure in the high-pressure cell medium at room temperature. After the HPHT process, the PCD table was removed from the cobalt-cemented tungsten carbide substrate by grinding away the cobalt-cemented tungsten carbide substrate.

TABLE II

Selected Magnetic Properties of Several Conventional PCD Tables						
Example	Average Diamond Particle Size (μm)	Sintering Pressure (GPa)	Specific Magnetic Saturation ($\text{G} \cdot \text{cm}^3/\text{g}$)	Calculated Co wt %	Coercivity (Oe)	Specific Permeability ($\text{G} \cdot \text{cm}^3/\text{g} \cdot \text{Oe}$)
15	20	4.61	19.30	9.605	94.64	0.2039
16	20	4.61	19.52	9.712	96.75	0.2018
17	20	4.61	19.87	9.889	94.60	0.2100
18	20	5.08	18.51	9.260	94.94	0.1990
19	20	5.08	18.71	9.351	100.4	0.1814
20	20	5.86	15.97	8.452	108.3	0.1567
21	20	4.61	17.17	8.543	102.0	0.1683
22	20	4.61	17.57	8.745	104.9	0.1675
23	20	5.08	15.10	8.014	111.2	0.1448
24	20	5.08	15.79	8.357	107.1	0.1568

As shown in Tables I and II, the conventional PCD tables listed in Table II exhibit a higher cobalt content therein than the PCD tables listed in Table I as indicated by the relatively higher specific magnetic saturation values. Additionally, the conventional PCD tables listed in Table II exhibit a lower coercivity indicative of a relatively greater mean free path between diamond grains, and thus may indicate relatively less diamond-to-diamond bonding between the diamond grains. Thus, the PCD tables according to examples of the invention listed in Table I may exhibit significantly less cobalt therein and a lower mean free path between diamond grains than the PCD tables listed in Table II.

18

Table III below lists conventional PCD tables that were obtained from PDC's. Each PCD table listed in Table III was separated from a cobalt-cemented tungsten carbide substrate bonded thereto by grinding.

TABLE III

Selected Magnetic Properties of Several Conventional PCD Tables				
Example	Specific Magnetic Saturation ($\text{G} \cdot \text{cm}^3/\text{g}$)	Calculated Co wt %	Coercivity (Oe)	Specific Permeability ($\text{G} \cdot \text{cm}^3/\text{g} \cdot \text{Oe}$)
25	17.23	8.572	140.4	0.1227
26	15.36	7.591	150.2	0.1059
27	15.19	7.560	146.1	0.1040
28	17.50	8.610	143.2	0.1208
29	17.13	8.525	152.1	0.1126
30	17.30	8.458	142.5	0.1193

TABLE III-continued

Selected Magnetic Properties of Several Conventional PCD Tables				
Example	Specific Magnetic Saturation ($\text{G} \cdot \text{cm}^3/\text{g}$)	Calculated Co wt %	Coercivity (Oe)	Specific Permeability ($\text{G} \cdot \text{cm}^3/\text{g} \cdot \text{Oe}$)
31	17.08	8.498	147.2	0.1160
32	15.10	8.011	144.1	0.1117

US 8,616,306 B2

19

Table IV below lists conventional PCD tables that were obtained from PDC's. Each PCD table listed in Table IV was separated from a cobalt-cemented tungsten carbide substrate bonded thereto by grinding the substrate away. Each PCD table listed in Table IV and tested had a leached region from which cobalt was depleted and an unleached region in which cobalt is interstitially disposed between bonded diamond grains. The leached region was not removed. However, to determine the specific magnetic saturation and the coercivity of the unleached region of the PCD table having metal-solvent catalyst occupying interstitial regions therein, the leached region may be ground away so that only the unleached region of the PCD table remains. It is expected that the leached region causes the specific magnetic saturation to be lower and the coercivity to be higher than if the leached region was removed and the unleached region was tested.

TABLE IV

Selected Magnetic Properties of Several Conventional Leached PCD Tables				
Example	Specific Magnetic Saturation (G·cm ³ /per gram)	Calculated Co wt %	Coercivity (Oe)	Specific Permeability (G·cm ³ /per (Oe))
33	17.12	8.471	143.8	0.1191
34	15.67	6.777	137.3	0.09920
35	15.87	7.897	140.1	0.1133
36	12.35	6.443	145.5	0.0890
37	13.89	6.914	142.0	0.09787
38	13.35	6.945	146.9	0.09503
39	13.67	6.863	133.8	0.1022
40	12.80	6.369	146.3	0.08749

As shown in Tables I, III, and IV, the conventional PCD tables of Tables III and IV exhibit a higher cobalt content therein than the PCD tables listed in Table I as indicated by the relatively higher specific magnetic saturation values. This is believed by the inventors to be a result of the PCD tables listed in Tables III and IV being formed by sintering diamond particles having a relatively greater percentage of fine diamond particles than the diamond particle formulations used to fabricate the PCD tables listed in Table I.

Examples 41-120 tested four different substrate interfacial surface geometries to evaluate the effect of the interfacial surface area of the substrate. Twenty samples of each substrate interfacial surface geometry were tested. All of the PDC's in examples 41-120 were fabricated by placing a mass of diamond particles having an average diamond particle size of about 19 μm adjacent to a cobalt-cemented tungsten carbide substrate in a niobium container, placing the container in a high-pressure cell medium, and subjecting the high-pressure cell medium and the container therein to an HPHT process using an HPHT cubic press to form a PCD table bonded to the substrate. The surface area of each anvil of the HPHT press and the hydraulic line pressure used to drive the anvils were selected so that the sintering pressure was at least about 7.7 GPa. The temperature of the HPHT process was about 1400° C. The sintering pressure of 7.7 GPa refers to the pressure in the high-pressure cell medium at room temperature, and the actual sintering pressure at the sintering temperature of about 1400° C, is believed to be greater.

The interfacial surface for the substrate in the PDC's of examples 41-60 was a substantially planar interfacial surface having essentially no surface topography other than surface roughness. The interfacial surface for the substrate in the PDC's of examples 61-80 was similar to the interfacial surface 404 shown in FIG. 4A. The interfacial surface for the sub-

20

strate in the PDC's of Examples 81-100 was slightly convex with a plurality of radially and circumferentially equally-spaced cylindrical protrusions. The interfacial surface for the substrate in the PDC's of examples 101-120 was similar to the interfacial surface 504 shown in FIGS. 5A and 5B.

After fabricating the PDC's of examples 41-120, the substrate of each PDC was brazed to an extension cobalt-cemented tungsten carbide substrate. The braze alloy had a composition of about 25 wt % Au, about 10 wt % Ni, about 15 wt % Pd, about 13 wt % Mn, and about 37 wt % Cu. The brazing process was performed at a brazing temperature of about 1013° C. After the brazing process, the PDC's of examples 41-120 were individually examined using an optical microscope to determine if cracks were present in the PCD tables.

Table V below lists the substrate diameter, surface area of the interfacial surface of the substrates for each type of substrate geometry, the ratio of the interfacial surface area of the substrate to a flat interfacial surface of a substrate with the same diameter, and the number of PDC samples in which the PCD table cracked upon brazing to the extension cobalt-cemented tungsten carbide substrate. As shown in Table V, as the surface area of the interfacial surface of the substrate decreases, the prevalence of the PCD table cracking decreases upon brazing.

TABLE V

Effect of Substrate Interfacial Surface Area on PCD Table Cracking Upon Brazing					
Example	Substrate Diameter (in)	Interfacial Surface Area of Substrate (in ²)	Ratio	Number of Samples That Cracked When Brazed	
41-60	0.635	0.308	1.0	0	0
61-80	0.625	0.398	0.772	0	0
81-100	0.635	0.524	0.588	7 out of 20	7 out of 20
101-120	0.635	0.585	0.526	9 out of 20	9 out of 20

Embodiments of Applications for PCD and PDC's

The disclosed PCD and PDC' embodiments may be used in a number of different applications including, but not limited to, use in a rotary drill bit (FIGS. 6A and 6B), a thrust-bearing apparatus (FIG. 7), a radial bearing apparatus (FIG. 8), a subterranean drilling system (FIG. 9), and a wire-drawing die (FIG. 10). The various applications discussed above are merely some examples of applications in which the PCD and PDC' embodiments may be used. Other applications are contemplated, such as employing the disclosed PCD and PDC' embodiments in friction stir welding tools.

FIG. 6A is an isometric view and FIG. 6B is a top elevation view of an embodiment of a rotary drill bit 600. The rotary drill bit 600 includes at least one PDC' configured according to any of the previously described PDC' embodiments. The rotary drill bit 600 comprises a bit body 602 that includes radially and longitudinally extending blades 604 with leading faces 606, and a threaded pin connection 608 for connecting the bit body 602 to a drilling string. The bit body 602 defines a leading end structure for drilling into a subterranean formation by rotation about a longitudinal axis 610 and application of weight-on-bit. At least one PDC' cutting element, configured according to any of the previously described PDC' embodiments (e.g., the PDC' 300 shown in FIG. 3A), may be affixed to the bit body 602. With reference to FIG. 6B, a plurality of PDC's 612 are secured to the blades 604. For

US 8,616,306 B2

21

example, each PDC **612** may include a PCD table **614** bonded to a substrate **616**. More generally, the PDC's **612** may comprise any PDC disclosed herein, without limitation. In addition, if desired, in some embodiments, a number of the PDC's **612** may be conventional in construction. Also, circumferentially adjacent blades **604** define so-called junk slots **618** therebetween, as known in the art. Additionally, the rotary drill bit **600** may include a plurality of nozzle cavities **620** for communicating drilling fluid from the interior of the rotary drill bit **600** to the PDC's **612**.

FIGS. **6A** and **6B** merely depict an embodiment of a rotary drill bit that employs at least one cutting element comprising a PDC fabricated and structured in accordance with the disclosed embodiments, without limitation. The rotary drill bit **600** is used to represent any number of earth-boring tools or drilling tools, including, for example, core bits, roller-cone bits, fixed-cutter bits, eccentric bits, bicenter bits, reamers, reamer wings, or any other downhole tool including PDC's, without limitation.

The PCD and/or PDC's disclosed herein (e.g., the PDC **300** shown in FIG. **3A**) may also be utilized in applications other than rotary drill bits. For example, the disclosed PDC embodiments may be used in thrust-bearing assemblies, radial bearing assemblies, wire-drawing dies, artificial joints, machining elements, and heat sinks.

FIG. **7** is an isometric cutaway view of an embodiment of a thrust-bearing apparatus **700**, which may utilize any of the disclosed PDC embodiments as bearing elements. The thrust-bearing apparatus **700** includes respective thrust-bearing assemblies **702**. Each thrust-bearing assembly **702** includes an annular support ring **704** that may be fabricated from a material, such as carbon steel, stainless steel, or another suitable material. Each support ring **704** includes a plurality of recesses (not labeled) that receive a corresponding bearing element **706**. Each bearing element **706** may be mounted to a corresponding support ring **704** within a corresponding recess by brazing, press-fitting, using fasteners, or another suitable mounting technique. One or more, or all of bearing elements **706** may be configured according to any of the disclosed PDC embodiments. For example, each bearing element **706** may include a substrate **708** and a PCD table **710**, with the PCD table **710** including a bearing surface **712**.

In use, the bearing surfaces **712** of one of the thrust-bearing assemblies **702** bear against the opposing bearing surfaces **712** of the other one of the bearing assemblies **702**. For example, one of the thrust-bearing assemblies **702** may be operably coupled to a shaft to rotate therewith and may be termed a "rotor." The other one of the thrust-bearing assemblies **702** may be held stationary and may be termed a "stator."

FIG. **8** is an isometric cutaway view of an embodiment of a radial bearing apparatus **800**, which may utilize any of the disclosed PDC embodiments as bearing elements. The radial bearing apparatus **800** includes an inner race **802** positioned generally within an outer race **804**. The outer race **804** includes a plurality of bearing elements **806** affixed thereto that have respective bearing surfaces **808**. The inner race **802** also includes a plurality of bearing elements **810** affixed thereto that have respective bearing surfaces **812**. One or more, or all of the bearing elements **806** and **810** may be configured according to any of the PDC embodiments disclosed herein. The inner race **802** is positioned generally within the outer race **804** and, thus, the inner race **802** and outer race **804** may be configured so that the bearing surfaces **808** and **812** may at least partially contact one another and move relative to each other as the inner race **802** and outer race **804** rotate relative to each other during use.

22

The radial bearing apparatus **800** may be employed in a variety of mechanical applications. For example, so-called "roller cone" rotary drill bits may benefit from a radial bearing apparatus disclosed herein. More specifically, the inner race **802** may be mounted to a spindle of a roller cone and the outer race **804** may be mounted to an inner bore formed within a cone and that such an outer race **804** and inner race **802** may be assembled to form a radial bearing apparatus.

Referring to FIG. **9**, the thrust-bearing apparatus **700** and/or radial bearing apparatus **800** may be incorporated in a subterranean drilling system. FIG. **9** is a schematic isometric cutaway view of a subterranean drilling system **900** that includes at least one of the thrust-bearing apparatuses **700** shown in FIG. **7** according to another embodiment. The subterranean drilling system **900** includes a housing **902** enclosing a downhole drilling motor **904** (i.e., a motor, turbine, or any other device capable of rotating an output shaft) that is operably connected to an output shaft **906**. A first thrust-bearing apparatus **700₁** (FIG. **7**) is operably coupled to the downhole drilling motor **904**. A second thrust-bearing apparatus **700₂** (FIG. **7**) is operably coupled to the output shaft **906**. A rotary drill bit **908** configured to engage a subterranean formation and drill a borehole is connected to the output shaft **906**. The rotary drill bit **908** is shown as a roller cone bit including a plurality of roller cones **910**. However, other embodiments may utilize different types of rotary drill bits, such as a so-called "fixed cutter" drill bit shown in FIGS. **6A** and **6B**. As the borehole is drilled, pipe sections may be connected to the subterranean drilling system **900** to form a drill string capable of progressively drilling the borehole to a greater depth within the earth.

A first one of the thrust-bearing assemblies **702** of the thrust-bearing apparatus **700** is configured as a stator that does not rotate and a second one of the thrust-bearing assemblies **702** of the thrust-bearing apparatus **700** is configured as a rotor that is attached to the output shaft **906** and rotates with the output shaft **906**. The on-bottom thrust generated when the drill bit **908** engages the bottom of the borehole may be carried, at least in part, by the first thrust-bearing apparatus **700₁**. A first one of the thrust-bearing assemblies **702** of the thrust-bearing apparatus **700₂** is configured as a stator that does not rotate and a second one of the thrust-bearing assemblies **702** of the thrust-bearing apparatus **700₂** is configured as a rotor that is attached to the output shaft **906** and rotates with the output shaft **906**. Fluid flow through the power section of the downhole drilling motor **904** may cause what is commonly referred to as "off-bottom thrust," which may be carried, at least in part, by the second thrust-bearing apparatus **700₂**.

In operation, drilling fluid may be circulated through the downhole drilling motor **904** to generate torque and effect rotation of the output shaft **906** and the rotary drill bit **908** attached thereto so that a borehole may be drilled. A portion of the drilling fluid may also be used to lubricate opposing bearing surfaces of the bearing elements **706** of the thrust-bearing assemblies **702**.

FIG. **10** is a side cross-sectional view of an embodiment of a wire-drawing die **1000** that employs a PDC **1002** fabricated in accordance with the teachings described herein. The PDC **1002** includes an inner, annular PCD region **1004** comprising any of the PCD tables described herein that is bonded to an outer cylindrical substrate **1006** that may be made from the same materials as the substrate **302** shown in FIG. **3A**. The PCD region **1004** also includes a die cavity **1008** formed therethrough configured for receiving and shaping a wire

US 8,616,306 B2

23

being drawn. The wire-drawing die **1000** may be encased in a housing (e.g., a stainless steel housing), which is not shown, to allow for handling.

In use, a wire **1010** of a diameter d_1 is drawn through die cavity **1008** along a wire drawing axis **1012** to reduce the diameter of the wire **1010** to a reduced diameter d_2 .

While various aspects and embodiments have been disclosed herein, other aspects and embodiments are contemplated. The various aspects and embodiments disclosed herein are for purposes of illustration and are not intended to be limiting. Additionally, the words “including,” “having,” and variants thereof (e.g., “includes” and “has”) as used herein, including the claims, shall have the same meaning as the word “comprising” and variants thereof (e.g., “comprise” and “comprises”).

The invention claimed is:

1. A polycrystalline diamond compact, comprising:
a substrate; and
a polycrystalline diamond table including a first polycrystalline diamond layer bonded to the substrate and at least a second polycrystalline diamond layer, at least an un-leached portion of the polycrystalline diamond table including:
a plurality of diamond grains defining a plurality of interstitial regions; and
a metal-solvent catalyst occupying at least a portion of the plurality of interstitial regions;
wherein the plurality of diamond grains and the metal-solvent catalyst collectively exhibit a coercivity of about 115 Oe or more and a specific magnetic saturation of about 15 G·cm³/g or less; and
wherein at least one of the second polycrystalline diamond layer exhibits a second average diamond grain size that is less than a first average diamond grain size of the first polycrystalline diamond layer or the first polycrystalline diamond layer includes a tungsten-containing material therein.
2. The polycrystalline diamond compact of claim 1 wherein the second average diamond grain size is about 90% to about 98% of the first average diamond grain size.
3. The polycrystalline diamond compact of claim 1 wherein the second average diamond grain size is about 90% to about 95% of the first average diamond grain size.
4. The polycrystalline diamond compact of claim 1 wherein the tungsten-containing material comprises tungsten carbide that is present in the first polycrystalline diamond layer in an amount of about 15 weight % or less.
5. The polycrystalline diamond compact of claim 4 wherein the amount is about 1 weight % to about 10 weight %.
6. The polycrystalline diamond compact of claim 1 wherein the first polycrystalline diamond layer does not include the tungsten-containing material.
7. The polycrystalline diamond compact of claim 1 wherein the second average diamond grain size of the second polycrystalline diamond layer is less than the first average diamond grain size of the first polycrystalline diamond layer and the first polycrystalline diamond layer includes the tungsten-containing material therein.
8. The polycrystalline diamond compact of claim 1 wherein the second average diamond grain size is about 20 μ m or less.
9. The polycrystalline diamond compact of claim 1 wherein the metal-solvent content is present in the at least the un-leached portion in an amount greater than 0 weight % to about 6 weight %.
10. The polycrystalline diamond compact of claim 9 wherein the amount is about 3 weight % to about 6 weight %.

24

11. The polycrystalline diamond compact of claim 1 wherein the substrate comprises an interfacial surface bonded to the first polycrystalline diamond layer, the interfacial surface including a plurality of protrusions, a ratio of a surface area of the interfacial surface in the absence of the plurality of protrusions to a surface area of the interfacial surface with the plurality of protrusions being about 0.650 to about 0.950.

12. The polycrystalline diamond compact of claim 1 wherein the coercivity is about 155 Oe to about 175 Oe, and wherein the specific magnetic saturation is about 10 G·cm³/g to about 15 G·cm³/g.

13. The polycrystalline diamond compact of claim 1 wherein the substrate comprises tungsten carbide, chromium carbide, or combinations thereof.

14. A rotary drill bit comprising a bit body including a leading end structure configured to facilitate drilling a subterranean formation, and a plurality of cutting elements mounted to the blades, at least one of the plurality of cutting elements configured as the polycrystalline diamond compact of claim 1.

15. A polycrystalline diamond compact, comprising:
a substrate; and
a polycrystalline diamond table including a first polycrystalline diamond layer bonded to the substrate and at least a second polycrystalline diamond layer, the second polycrystalline diamond layer exhibiting a second average diamond grain size that is less than a first average diamond grain size of the first polycrystalline diamond layer, at least an un-leached portion of the polycrystalline diamond table including:
a plurality of diamond grains defining a plurality of interstitial regions;
a metal-solvent catalyst occupying at least a portion of the plurality of interstitial regions; and
wherein the plurality of diamond grains and the metal-solvent catalyst collectively exhibit a coercivity of about 115 Oe or more and a specific magnetic saturation of about 15 G·cm³/g or less.

16. The polycrystalline diamond compact of claim 15 wherein the second average diamond grain size is about 90% to about 98% of the first average diamond grain size.

17. The polycrystalline diamond compact of claim 15 wherein the second average diamond grain size is about 20 μ m or less.

18. The polycrystalline diamond compact of claim 15 wherein the metal-solvent content is present in the at least the un-leached portion in an amount of about 3 weight % to about 6 weight %.

19. The polycrystalline diamond compact of claim 15 wherein the first polycrystalline diamond layer comprises a tungsten-containing material therein present in an amount of about 15 weight % or less.

20. The polycrystalline diamond compact of claim 15 wherein the substrate comprises an interfacial surface bonded to the first polycrystalline diamond layer, the interfacial surface including a plurality of protrusions, a ratio of a surface area of the interfacial surface in the absence of the plurality of protrusions to a surface area of the interfacial surface with the plurality of protrusions being about 0.650 to about 0.950.

21. The polycrystalline diamond compact of claim 15 wherein the substrate comprises tungsten carbide, chromium carbide, or combinations thereof.

22. A rotary drill bit comprising a bit body including a leading end structure configured to facilitate drilling a subterranean formation, and a plurality of cutting elements

US 8,616,306 B2

25

mounted to the blades, at least one of the plurality of cutting elements configured as the polycrystalline diamond compact of claim 15.

23. A polycrystalline diamond compact, comprising:
a substrate; and
a polycrystalline diamond table including a first polycrystalline diamond layer bonded to the substrate and at least a second polycrystalline diamond layer, the first polycrystalline diamond layer including a tungsten-containing material therein, at least an un-leached portion of the polycrystalline diamond table including:
a plurality of diamond grains defining a plurality of interstitial regions;
a metal-solvent catalyst occupying at least a portion of the plurality of interstitial regions; and
wherein the plurality of diamond grains and the metal-solvent catalyst collectively exhibit a coercivity of about 115 Oe or more and a specific magnetic saturation of about 15 G·cm³/g or less.

24. The polycrystalline diamond compact of claim 23 wherein the tungsten-containing material comprises tungsten carbide that is present in the first polycrystalline diamond layer in an amount of about 15 weight % or less.

25. The polycrystalline diamond compact of claim 24 wherein the amount is about 1 weight % to about 10 weight %.

26. The polycrystalline diamond compact of claim 23 wherein the second polycrystalline diamond layer exhibits a

26

second average grain size is less than a first average diamond grain size of the first polycrystalline diamond layer.

27. The polycrystalline diamond compact of claim 23 wherein the second average diamond grain size is about 20 μm or less.

28. The polycrystalline diamond compact of claim 23 wherein the metal-solvent content is present in the at least the un-leached portion in an amount of about 3 weight % to about 6 weight %.

29. The polycrystalline diamond compact of claim 23 wherein the substrate comprises an interfacial surface bonded to the first polycrystalline diamond layer, the interfacial surface including a plurality of protrusions, a ratio of a surface area of the interfacial surface in the absence of the plurality of protrusions to a surface area of the interfacial surface with the plurality of protrusions being about 0.650 to about 0.950.

30. The polycrystalline diamond compact of claim 23 wherein the substrate comprises tungsten carbide, chromium carbide, or combinations thereof.

31. A rotary drill bit comprising a bit body including a leading end structure configured to facilitate drilling a subterranean formation, and a plurality of cutting elements mounted to the blades, at least one of the plurality of cutting elements configured as the polycrystalline diamond compact of claim 23.

* * * * *